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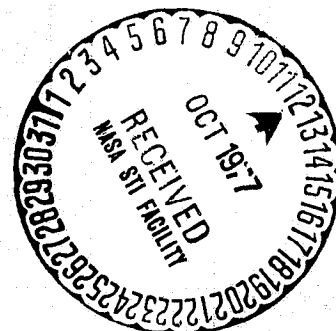
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CORRELATION OF CREEP RATE WITH MICROSTRUCTURAL CHANGES DURING HIGH TEMPERATURE CREEP

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CORRELATION OF CREEP RATE WITH MICROSTRUCTURAL CHANGES DURING HIGH TEMPERATURE CREEP

ABSTRACT

In future designs of subsonic and hypersonic aircraft and space vehicles, many structural metallic components will be designed to withstand repeated applications of complex stress cycles at elevated temperatures and will generally be required to experience less than 0.5% creep strain during the design life. Consequently, the primary stage of creep is of significant importance. It is known that the course of primary creep can be favorably altered by the use of various thermomechanical treatments to produce more creep-resistant substructures, but our understanding of how the stability of these structures during primary creep is related to creep rate is limited.

In this research, creep tests have been conducted on Haynes 188 cobalt-base alloy and alpha titanium. The tests on Haynes 188 were conducted at 1600° and 1800°F for stresses from 3 to 20 ksi, and the as-received, mill-annealed results were compared to specimens given 5%, 10%, and 15% room temperature prestrains and then annealed one hour at 1800°F. The tests on alpha titanium were performed at 7250 and 10,000 psi at 500°C. One creep test was done at 527°C and 10,000 psi to provide information on kinetics. Results for annealed titanium were compared to specimens given 10% and 20% room temperature prestrains followed by 100 hours recovery at 550°C. Electron microscopy was used to relate dislocation and precipitate structure to the creep behavior of the two materials.

The results on Haynes 188 alloy reveal that the time to reach 0.5% creep strain at 1600°F increases with increasing prestrain for exposure times less than 1000 hours, the increase at 15% prestrain being more than a factor of ten. At exposure times of 100 hours this corresponds to a

60% increase in design stress. Above 1000 hours at 1600°F, the beneficial effects of substructure on primary creep rate are the result of heterogeneous precipitation of $M_{23}C_6$ carbides on dislocation substructures produced by the prestrain, thus stabilizing these structures during primary creep. The disappearance of the beneficial effects for long creep exposures has been related to deformation-enhanced kinetics for coarsening of grain boundary precipitates during creep, presumably $M_{23}C_6$, which leads to grain boundary instability under stress and subsequent grain boundary migration and grain growth.

The creep results for alpha titanium have shown that the use of a prior thermomechanical treatment consisting of a 10% room temperature prestrain and recovery of 100 hours at 550°C increased the steady-state creep rate, even though clear subgrains bounded by arrays of dislocations had been produced. The creep rate was decreased, however, when a prior 20% room temperature prestrain was used without recovery to produce a tangled dislocation structure. It was concluded that the creep of alpha titanium at 7250 and 10,000 psi is controlled by the availability of mobile dislocations and that the well-defined subgrains produced in this study acted as a ready source of mobile dislocations due to the occurrence of long segments of parallel, unpinned dislocations on {1120} planes. The creep curves obtained for annealed titanium exhibited a minimum creep rate followed by a gradual increase to an apparent steady-state value. This gradual increase was accompanied by subgrain formation, and it was concluded that the true steady-state rate was that approached during this subgrain formation. The activation energy for such steady-state creep was found to be about 28,800 calories per mole and the stress exponent was found to be about five.

ACKNOWLEDGMENTS

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CORRELATION OF CREEP RATE WITH MICROSTRUCTURAL CHANGES DURING HIGH TEMPERATURE CREEP

1. PREFACE

In many aerospace structural components where high temperature creep is the principal limitation to design life, no more than 0.5% of creep strain can be tolerated. The early stages of creep are therefore of primary importance. Although it is known that the nature of primary creep can be favorably altered by thermomechanical treatments, we have only limited understanding of the stability of substructures produced and how this relates to creep rate.

In this research, the techniques of electron microscopy were used to examine the microstructural changes which occur during primary creep for two important engineering materials: (1) unalloyed titanium, and (2) the cobalt-base alloy, Haynes 188. The following two sections of this report present these results. The three appendices attached present: (1) a summary of the experimental approach developed at NASA-Langley under this grant for thinning of Haynes 188 for transmission electron microscopy, (2) a paper prepared for submission to Metallurgical Transactions of the ASM-AIME on analyzing the accuracy of beam axis solutions from Kikuchi projections, and (3) a reprint of a recently published paper which describes a computer program for generation of divergent beam diffraction phenomena. The latter two papers were produced partly through support under the present NASA grant.

The results reported herein were obtained during the period from July 1, 1972 to February 29, 1976.

A fourth appendix presents factors for conversion of the units used in this report to the International System of Units (SI).

2. EFFECT OF THERMOMECHANICAL TREATMENTS ON THE CREEP OF HAYNES 188 ALLOY

INTRODUCTION

The effects of the external variables of stress and temperature on the steady-state creep behavior of pure metals and dilute alloys at elevated temperatures has been extensively studied and an understanding of these effects is gradually emerging⁽¹⁻³⁾. For example, the temperature dependence of creep is now believed to be simply related to self-diffusion and the effect of stress on creep rate can usually be expressed as a power law of exponent 4-6 for the normal range of stresses encountered in engineering service. More recently, the effects of the physical parameters of elastic modulus and stacking fault energy have also been included in creep correlations^(1,2).

The important effects of microstructure on creep behavior, however, are still not well understood. For example, the initial creep rate of high purity aluminum may be decreased by more than four orders of magnitude, for the same stress and temperature, simply by changing the initial microstructure from the as-annealed state to a structure containing subgrains bounded by dislocation networks formed by prior thermomechanical treatments⁽⁴⁾.

In future designs of aircraft and space vehicles, many structural metallic components will be designed to withstand repeated applications of complex stress cycles at elevated temperatures. Since these metals will generally be required to experience less than 0.5% creep strain during their design life, it is clear that significant attention needs to be given to the microstructural changes which occur during primary creep, since 0.5%

creep strain generally falls within the primary stage. It is therefore anticipated that significant improvement of design life can be obtained in high temperature alloys by development of improved initial dislocation and precipitate structures through appropriate thermomechanical treatments.

The purpose of this investigation was to examine the relation of creep rate to microstructure during primary creep for an alloy which shows significant promise in high temperature structural applications and for which the superimposed effects of dislocation structure and finely dispersed precipitates could be studied. For these reasons, the cobalt-base alloy Haynes 188 was chosen. The effects of dispersed precipitates were considered to be important since variations in creep rate for Al-0.5 at % Ag have recently been related to the effects of pinning of dislocation substructure by γ' precipitates and their gradual growth during creep straining, and these effects apparently account for unusual temperature and stress dependence of the creep rate^(5,6). In the case of Haynes 188, the dispersed phases available are predominantly two types of carbides, M_6C and $M_{23}C_6$. The matrix is stabilized in the FCC structure by nickel, iron, carbon, and manganese^(7,8). The M_6C carbides are found dispersed in the matrix after annealing at 2150°F, and further exposure at temperatures above about 1650°F tends to produce additional precipitation of M_6C . Exposure between 1300° and 1650°F, however, tends to produce the complex chromium-nickel-cobalt carbide, $M_{23}C_6$. This $M_{23}C_6$ carbide appears to be heterogeneously nucleated, and offers significant promise for improved creep strength during primary creep by interaction with dislocation structures produced by thermomechanical means.

In this study, dislocation substructures were introduced by room temperature true tensile prestrains of 5, 10, and 15%. These substructures were then modified by heating in air for one hour at 1800^oF prior to creep testing. Results were compared to those for specimens given no prestrain to determine the effects of the thermomechanical treatments employed.

EXPERIMENTAL PROCEDURE

The material used in this study was obtained from the Stellite Division of the Cabot Corporation. The nominal composition of this alloy is 20-24% Cr, 20-24% Ni, 13-16% W, 0.05-0.15% C, 0.03-0.15% La, 0.20-0.50% Si, 1.25% Mn, up to 3% Fe, with the balance cobalt. The sheet was received in the mill annealed condition and had been stretch flattened 1-2%. The 0.2% offset yield strength was found to average about 78 ksi, compared with that specified by the producer to be 67 ksi for annealed sheet. It is believed that this difference is due to the cold work produced during stretch flattening. Creep specimens were machined into the configuration shown in Figure 2.1. Some specimens were strained to 5, 10, and 15% true tensile strain using an Instron machine. In the present study, creep specimens were given a preoxidation treatment in air at 1800°F for one hour. The primary purpose of this preoxidation treatment was to form a layer of oxide which provides for high surface emittance, a condition required for the material to be used as a thermal protection shield in certain space applications. Creep specimens were placed in the creep furnace for three to four hours prior to creep straining to allow them to stabilize at the desired creep temperature.

In view of the various thermomechanical treatments which were to be used to generate stable substructures prior to creep tests, an investigation was made to determine if the one hour preoxidation treatment at 1800°F also could serve as a flow stress recovery treatment and provide

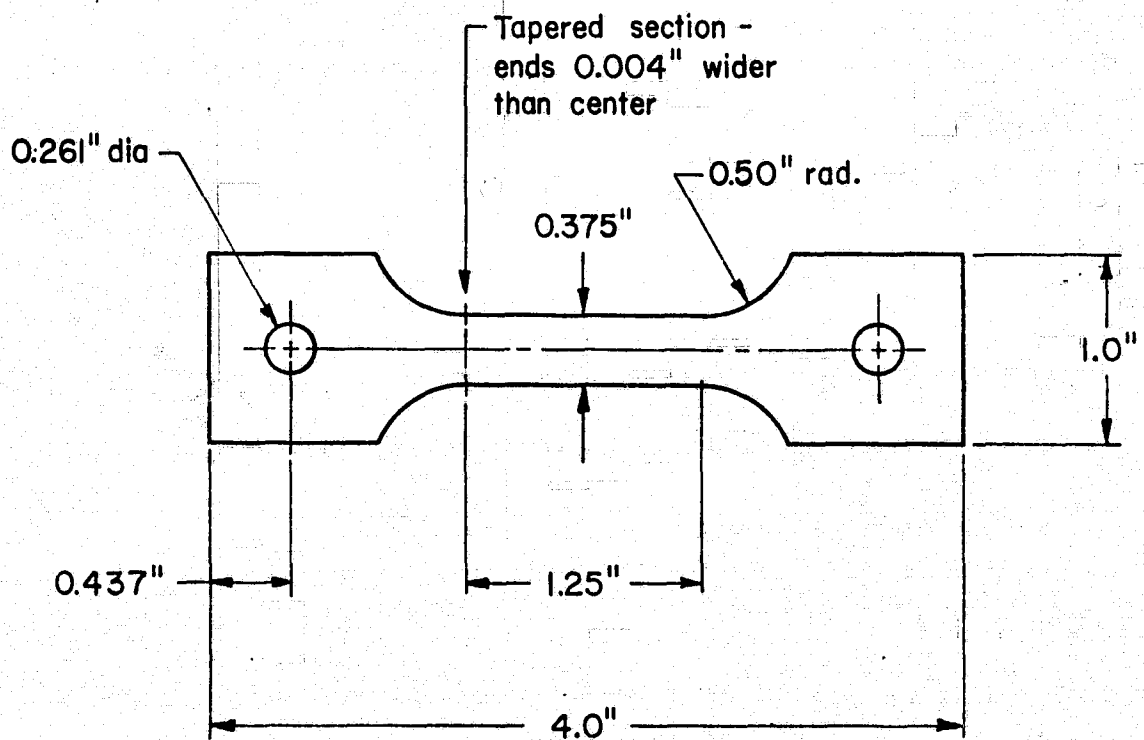


Figure 2.1. Creep and tensile test specimen configuration.

a nearly stable dislocation structure when conducted following the 10% prestrain. For this purpose, the room temperature 0.2% offset yield strength of the alloy was measured after various recovery times at 1800°F in air and the results are shown in Figure 2.2. It is seen that the flow stress of the as-received alloy drops from 78 ksi to 64 ksi within one hour of annealing and stays at this value for at least 24 hours. The flow stress of the specimens prestrained 10% drops from 115 ksi to about 90 ksi within one hour and decreases slowly on further exposure to about 85 ksi in 24 hours. Apparently, age hardening does not occur for either the as-received or the prestrained alloys, at least for the first 24 hours. The decrease in yield strength during annealing is believed to be due to the rearrangement of dislocations which were introduced during cold working. The retention of increased yield strength of the prestrained alloy after the 24 hour preoxidation treatment is believed to be due to the metastable dislocation structures developed and perhaps to a favorable distribution of precipitate particles. It has been concluded that the one hour pre-oxidation treatment at 1800°F is sufficient to produce a nearly stable dislocation substructure following the room temperature prestrains used in this investigation.

Scanning electron micrographs at 1000X of polished and etched specimens of Haynes 188 are shown in Figure 2.3. Figure 2.3a shows the microstructure of the as-received, mill annealed, sheet; and Figure 2.3b shows the structure after 10% prestrain and the one hour preoxidation treatment at 1800°F. Note that the as-received sheet has no visible grain boundary precipitates; the white precipitates are reported to be M_6C

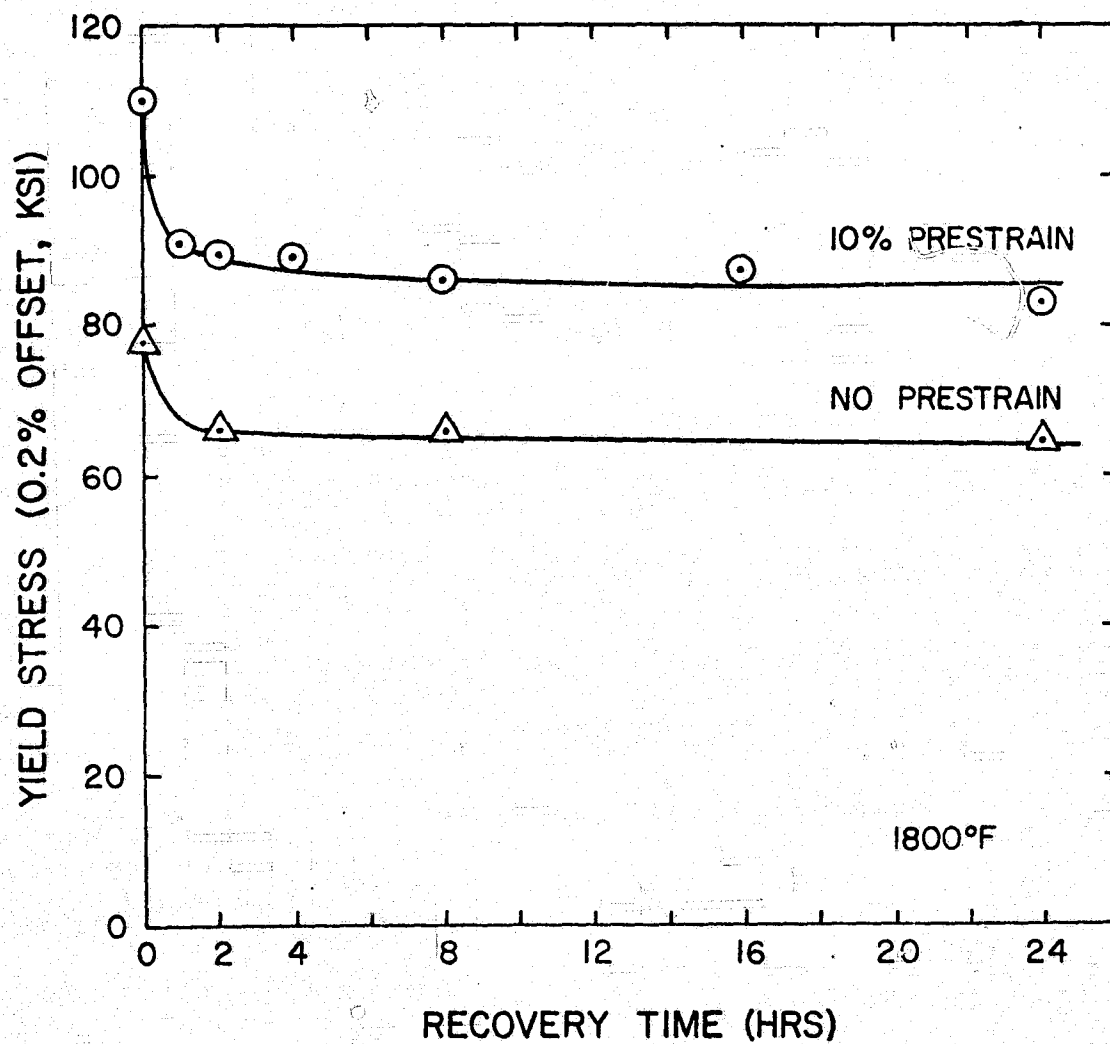
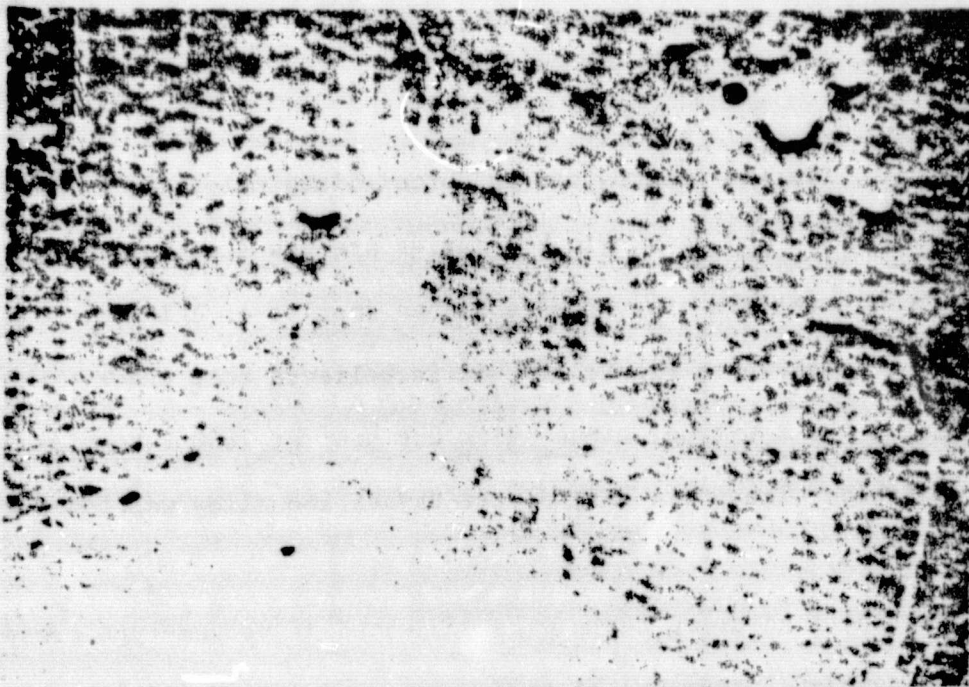
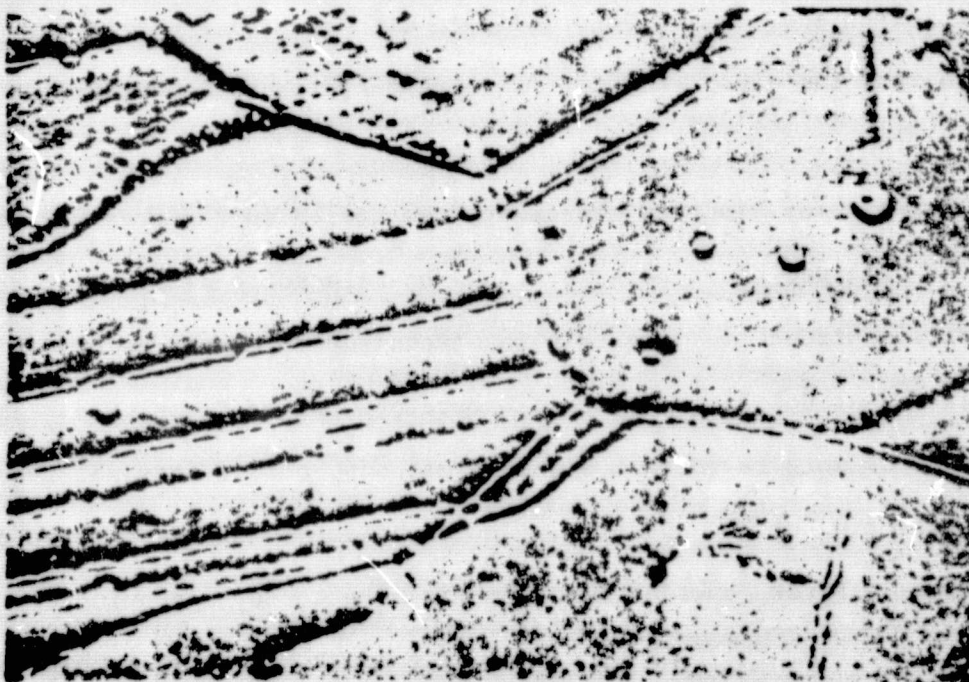


Figure 2.2. Flow stress recovery behavior of Haynes 188 at 1800 F.



(a) Mill annealed at 2150°F, stretch flattened.



(b) 10% prestrained, preoxidized one hour at 1800°F.

Figure 2.3. Scanning electron micrographs of polished etched Inconel 188 alloy. 1500X.

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carbides⁽⁸⁾. In the prestrained and preoxidized material, however, the grain boundaries and many twin boundaries are now seen to be decorated with precipitate. In view of established results of aging studies at 1800°F⁽⁸⁾, as shown in Figure 2.4, it is believed that these precipitates are also of the M_6C type, since aging one hour at 1800°F remains within the M_6C field. The grain size of the Haynes 188 alloy was found to be about 0.03 mm.

Constant load creep tests were conducted in vacuum-type creep apparatus at a pressure of 1 torr and at temperatures of 1600°F (871°C) and 1800°F (972°C). Creep stresses ranged from 3 ksi to 20 ksi. The temperature was maintained to $\pm 3^\circ\text{C}$. A platinum strip strain gauge was attached to each specimen and a microscope equipped with a vernier micrometer was used to read the creep strains. The creep strain so determined is accurate to ± 0.0003 .

Transmission electron microscopy was needed in the present study so that the observed creep behavior could be related to dislocation and precipitate structure. The thinning technique developed by Dubose and Stiegler⁽⁹⁾ at Oak Ridge National Laboratory has been employed. This technique is more rapid than standard methods, and it does not require the close attention of the operator. The technique consists of three separate operations. First, a wafer about 3mm in diameter by approximately 20 mils thick is cut from the bulk specimen using spark discharge machine. Next, a jet of electrolyte is used to produce flat-bottom dimples from both sides of the disc leaving an area about 2 mils thick surrounded by a thick ring of unpolished material. Finally, the dimpled disc specimen is

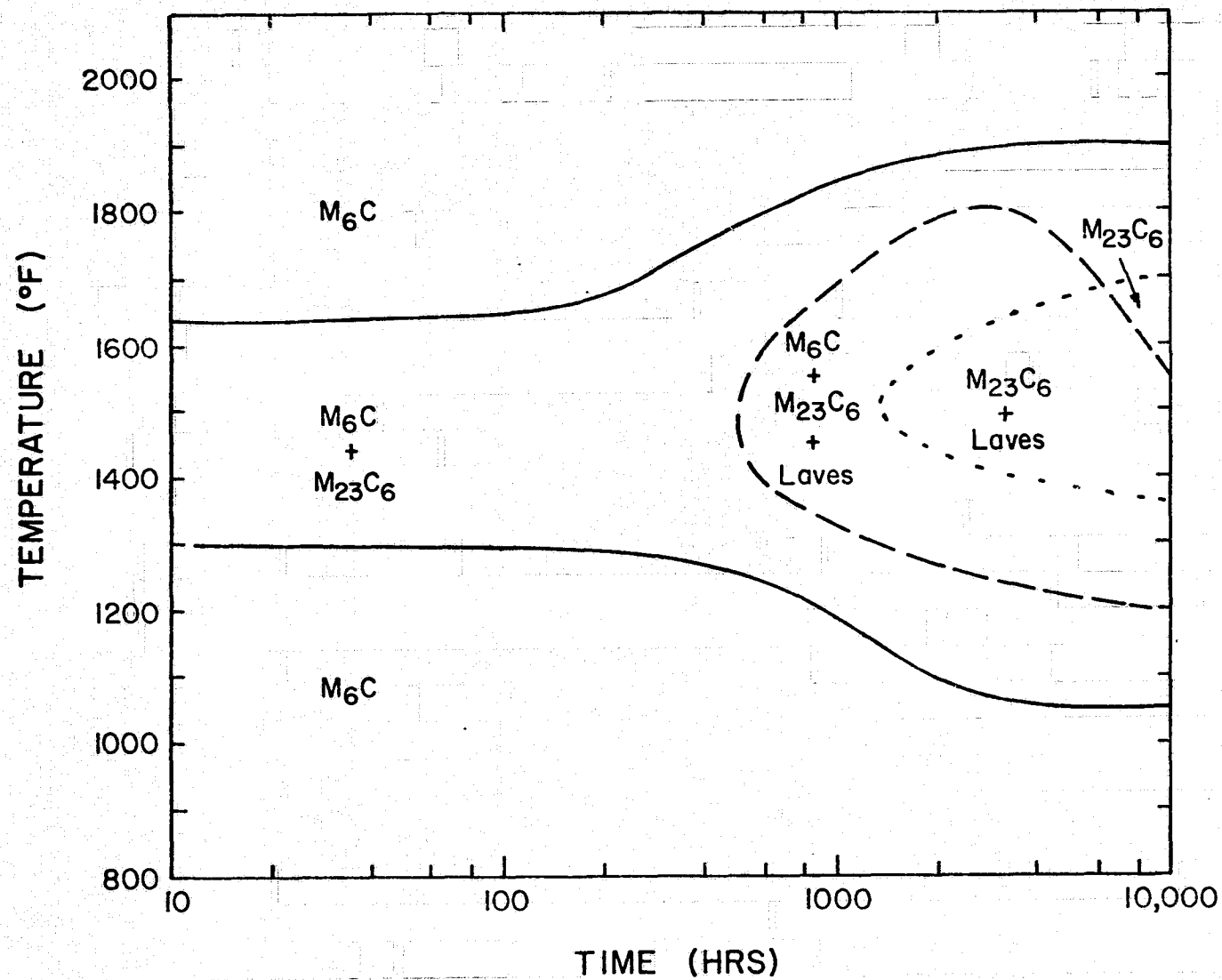


Figure 2.4. Structural reactions during aging of Haynes 188. Order of phases listed indicates relative abundance. (After Herchenroeder, Ref. 11)

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placed in a standard electropolishing dish where it is illuminated from one side by a focused light beam transmitted by a fiber-optics system. When a perforated hole is formed during polishing, the transmitted light activates a photo-cell detector which stops the polishing action.

The thinning apparatus basically includes: (1) a jet polisher, (2) a standard polishing dish, (3) a semi-automatic cooling system, (4) a D-C power supply with up to 600 volts output capacity, and (5) a light source and controller. This apparatus has been constructed and installed at Langley Research Center. A circulating ethylene glycol solution which can be cooled to -35°C by liquid nitrogen is used to cool the electrolytes in both thinning stages. Low operating temperature in the final stage of polishing is normally desired in order to eliminate any structural modification during thinning. Low temperature is also desired so that large uniform thin foils transparent to electrons can be more frequently obtained.

An electrolyte containing 33 cc. concentrated HNO_3 and 67 cc. methyl alcohol operating at room temperature and 300 milliamp was found to produce the best dimple configuration in Haynes 188. However, the dimples generated were not flat enough for final polishing. It was found later that the dimple configuration could be improved to a satisfactory shape if the operating temperature was lowered to about 0° to 5°C . The same solution used in jet polishing was also found to be satisfactory for use in the final stage of thinning.

A highly detailed discussion of the thinning apparatus and its use, as well as a listing of the various thinning solutions developed for use

on Haynes 188, is attached as Appendix A. Characterization of as-received and as-preoxidized structures by transmission electron microscopy is given in Figures 2.5 and 2.6. Figure 2.5 shows the as-received, mill annealed and stretch flattened condition. Note that considerable dislocation density is found near the twin boundaries, as previously suspected in discussion of Figure 2.2. Note that some stacking faults are visible. Figure 2.6 shows how the preoxidation treatment (1800°F, 1 hr) has decorated the twin boundaries with precipitate as also detected by scanning electron microscopy in Figure 2.3b. The dislocation density has also been reduced by the preoxidation treatment.

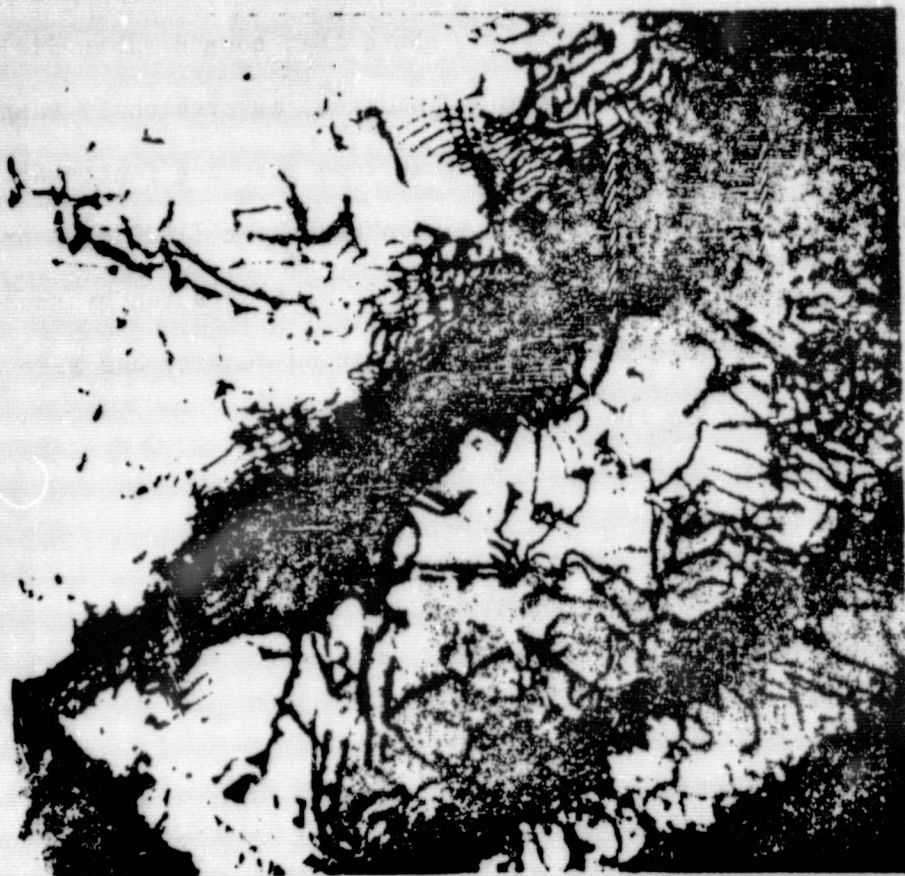


Figure 2.5. Transmission electron micrograph showing as-received, stretch-flattened condition for Haynes 188 alloy. 20,000X.



Figure 2.6. Transmission electron micrograph showing dislocation and precipitate structure of Haynes 188 alloy after preoxidation treatment for one hour at 1800°F. 28,000X

RESULTS AND DISCUSSION

Constant load creep tests were performed at one Torr for temperatures of 1600°F and 1800°F. In general, creep stresses of 3, 5, 10, 15, and 20 ksi were employed, the choice depending upon the temperature. Room temperature tensile prestrains of 5%, 10%, and 15% followed by heating in air for one hour at 1800°F were generally used for study of the effects of thermomechanical treatments and these were compared to specimens of as-received material which were tested without prestrain but which were given the preoxidation treatment for one hour in air at 1800°F.

Typical creep curves for Haynes 188 alloy are shown in Figures 2.7 through 2.10. In each case, the degree of prestrain used is shown; the symbol "A-R" indicates the as-received condition. An arrow at the end of the curve indicates that the test was discontinued without fracture; a cross mark indicates failure.

Examination of the creep curves reveals that in general a minimum creep rate was achieved following a primary stage, the minimum rate then being followed by increasing creep rate as the specimen proceeded toward eventual fracture. Several observations may be noted from the creep behavior in these tests:

1. For creep at 1600°F, minimum creep rate tended to decrease with increasing prestrain for all conditions tested. This behavior is shown graphically in Figure 2.11 where the logarithm of the minimum creep rate is shown as a function of the logarithm of the creep stress.

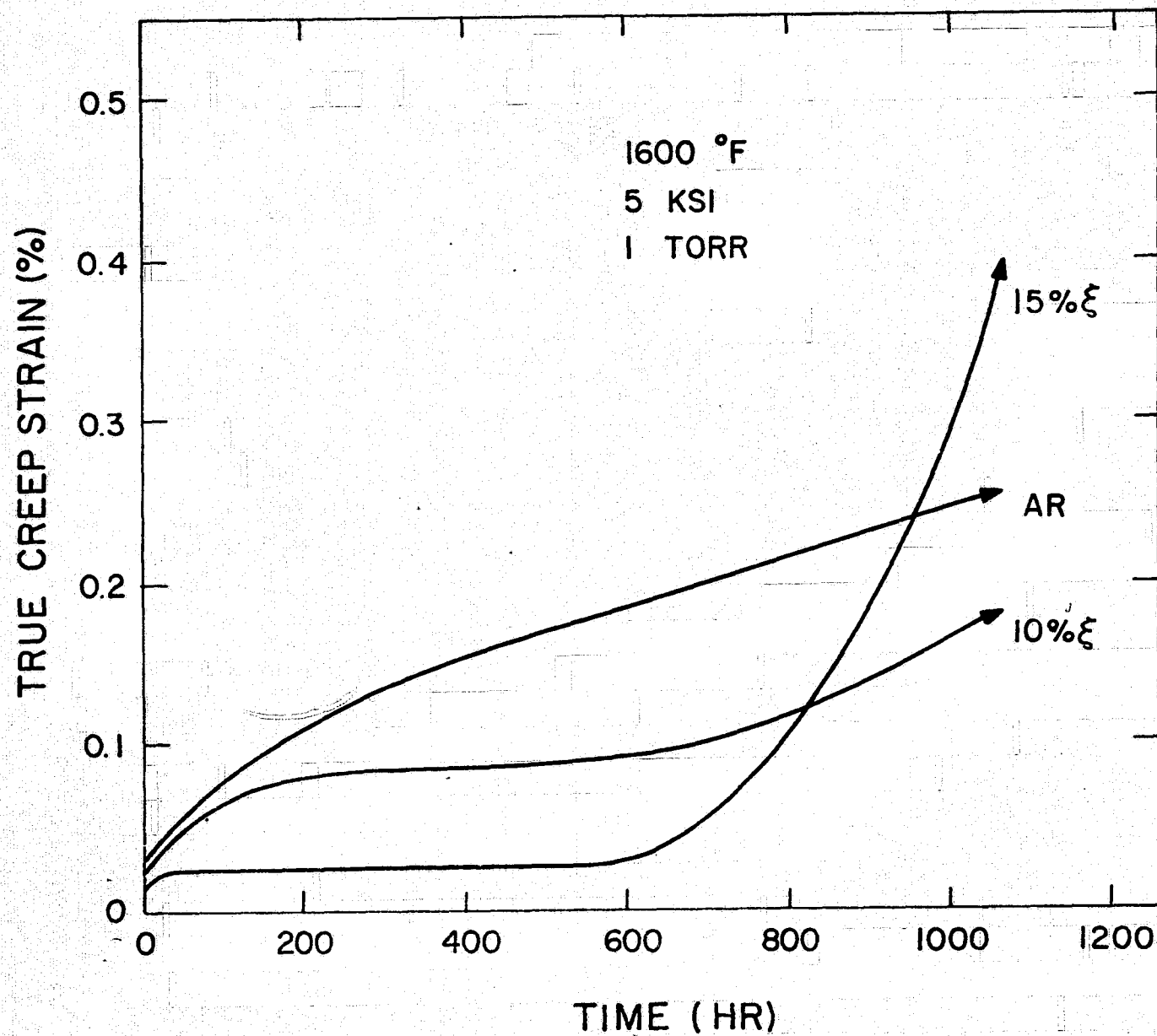


Figure 2.7 Creep curves for Haynes 188 alloy at 5 ksi and 1600°F.

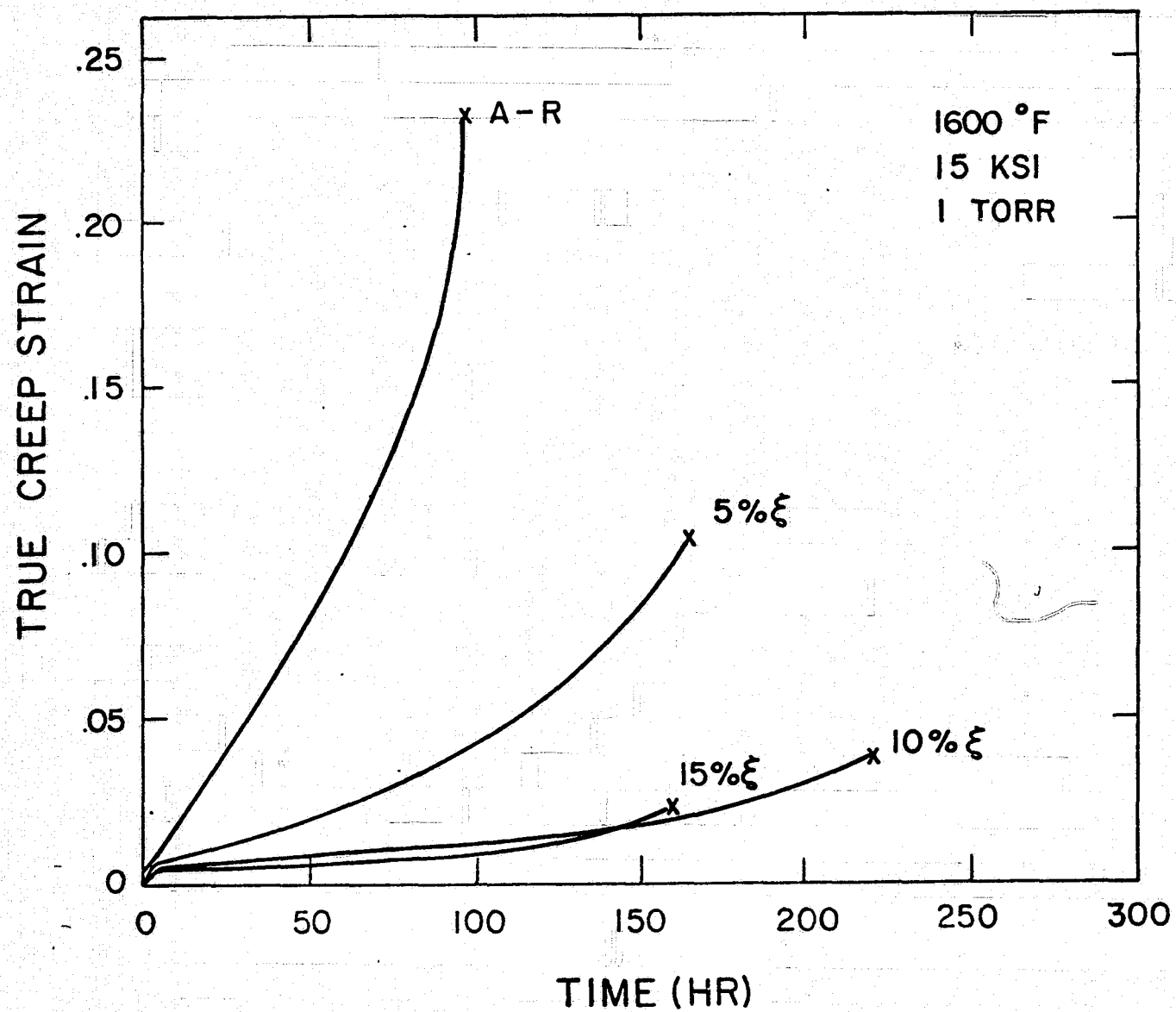


Figure 2.8 Creep curves for Haynes 188 alloy at 15 ksi and 1600°F.

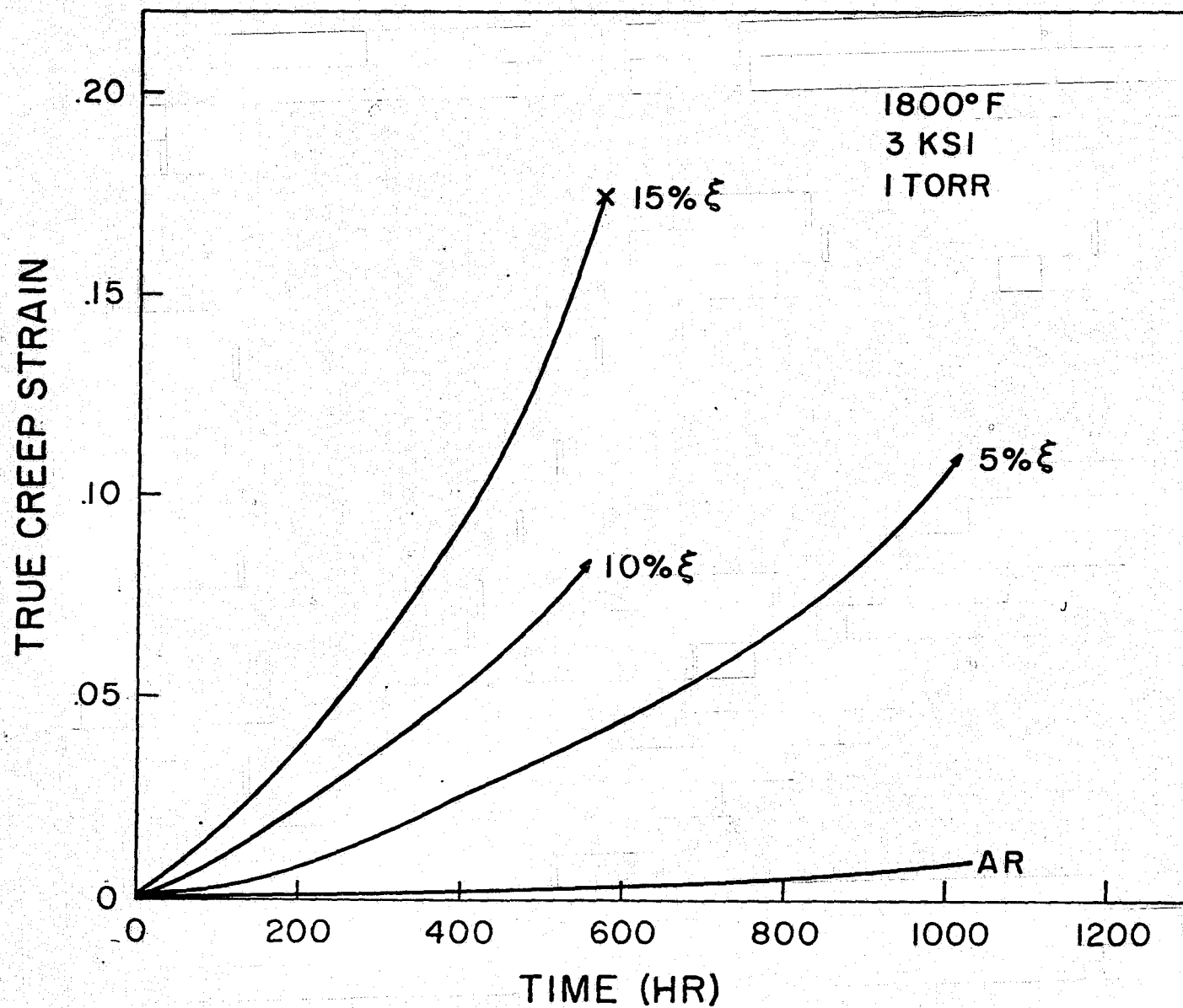


Figure 2.9 Creep curves for Haynes 188 alloy at 3 ksi and 1800°F.

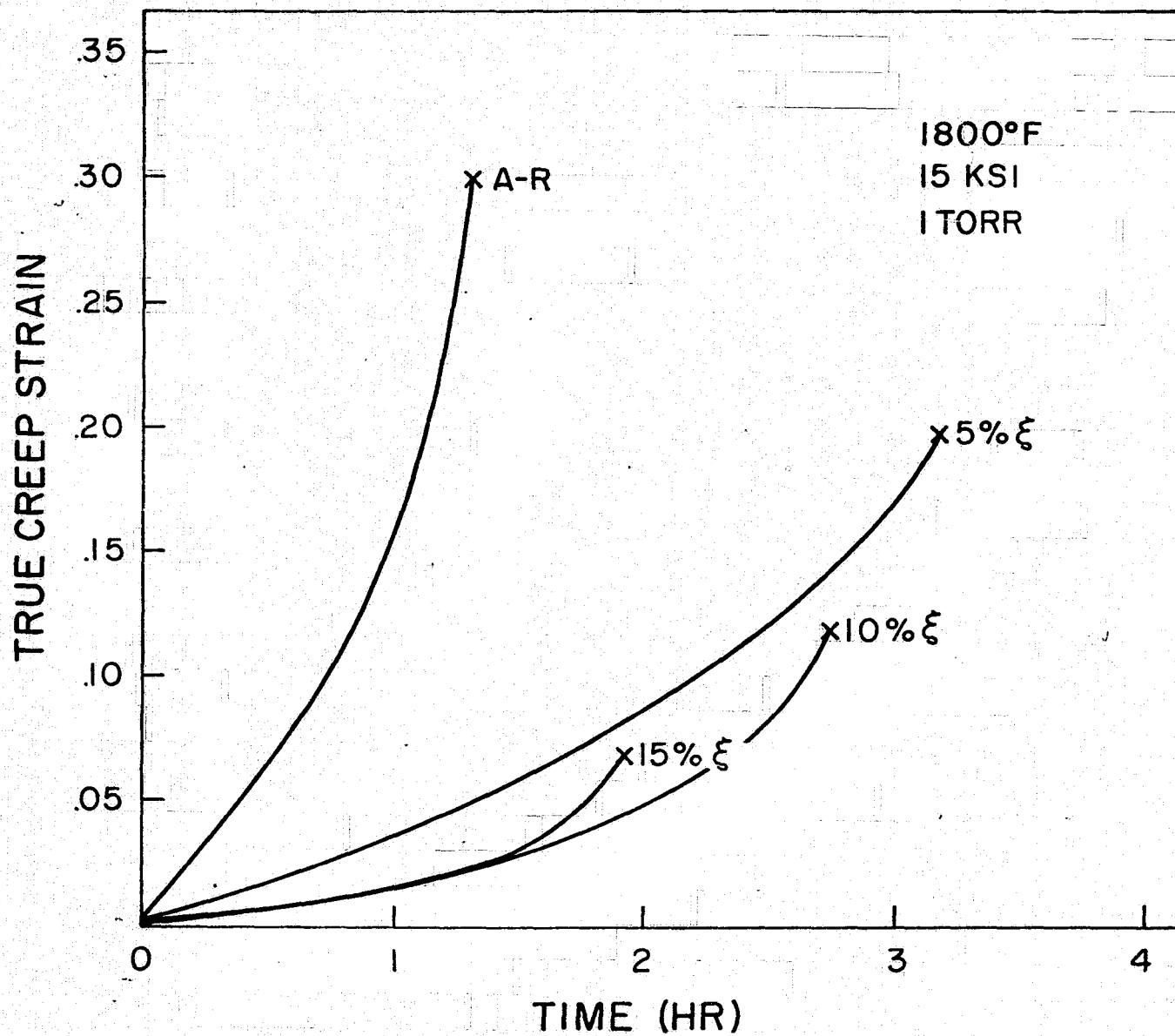


Figure 2.10 Creep curves for Haynes 188 alloy at 15 ksi and 1800°F.

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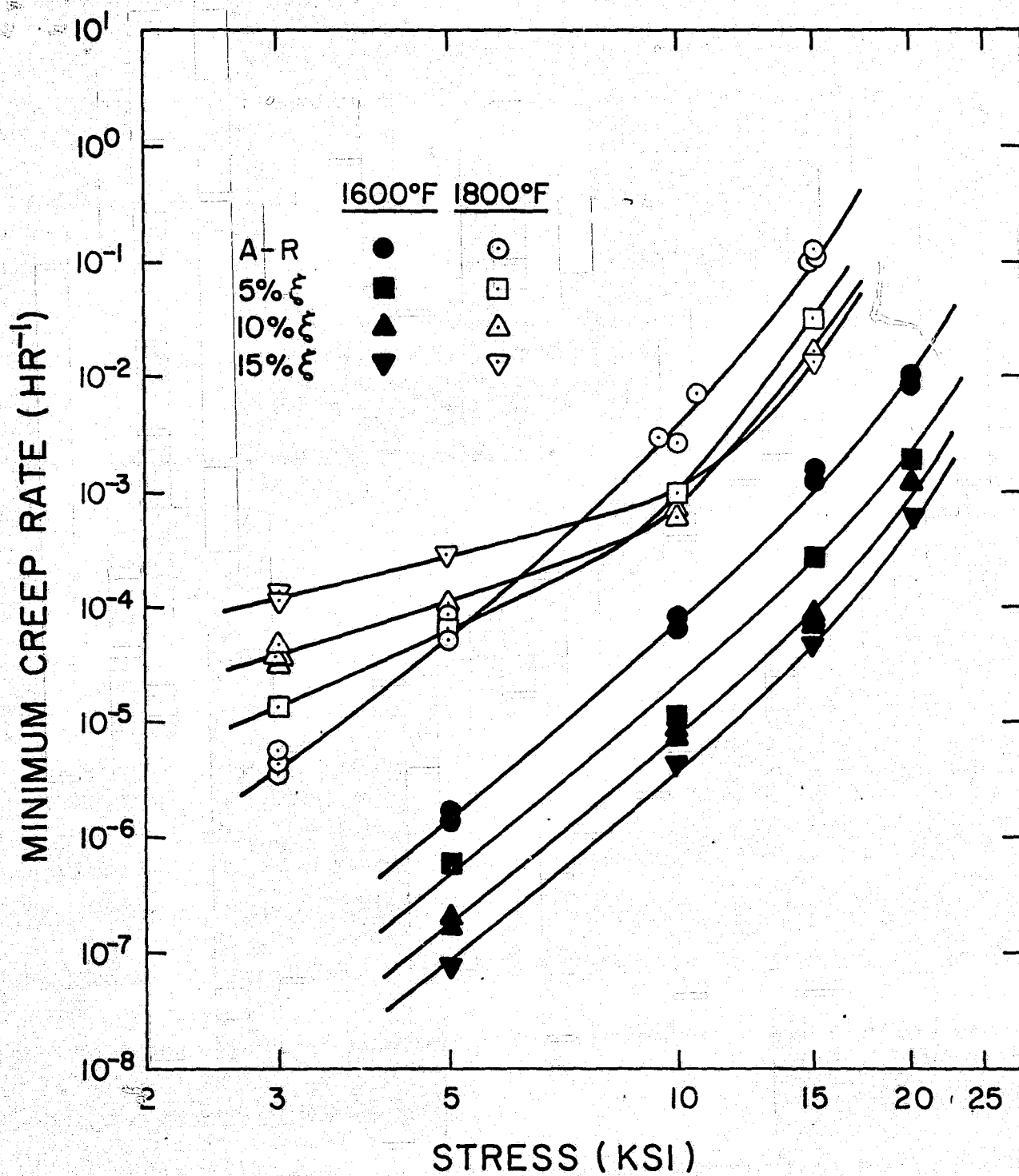


Figure 2.11 Minimum creep rate data for Haynes 188 alloy at various stresses.

2. For creep at 1800°F , two different types of behavior were found. At 10 and 15 ksi creep stress, minimum creep rate tended to decrease with increasing prestrain. At 3 and 5 ksi, the minimum creep rate tended to increase with increasing prestrain. This is also shown in Figure 2.11.

3. The creep strain at fracture tended to decrease with decreasing minimum creep rate.

Reference to the creep rate data summarized in Figure 2.11 reveals that minimum creep rates in Haynes 188 at 1600°F are reduced by a factor of 18 when 15% prestrain is used compared to as-received material. This reduction is relatively independent of the creep stress. At 1800°F , the minimum creep rate is about ten times lower for 15% prestrain at 15 ksi, but is about 30 times higher than as-received material at 3 ksi. This inversion of the effect of increasing prestrain is suggestive of substructural instability, since the lower stresses also mean longer exposure times at 1800°F . For example, minimum creep rates are achieved within one hour for creep at 1800°F and 15 ksi, but about fifty hours are needed when creep occurs at 1800°F and 3 ksi.

Since the time to reach 0.5% creep strain is an important feature of the present study, the logarithm of this parameter is shown in Figure 2.12 as a function of creep stress. It is apparent that an inversion occurs for both creep temperatures when time to 0.5% strain is plotted. For creep at 1600°F , significant improvement in creep behavior is demonstrated for exposure times less than about 1000 hours, with the greatest improvement for the highest prestrain. For example, for 100 hours exposure at 1600°F , a 60% increase in design stress could be tolerated when the thermomechanical treatment involving 15% prestrain is used.

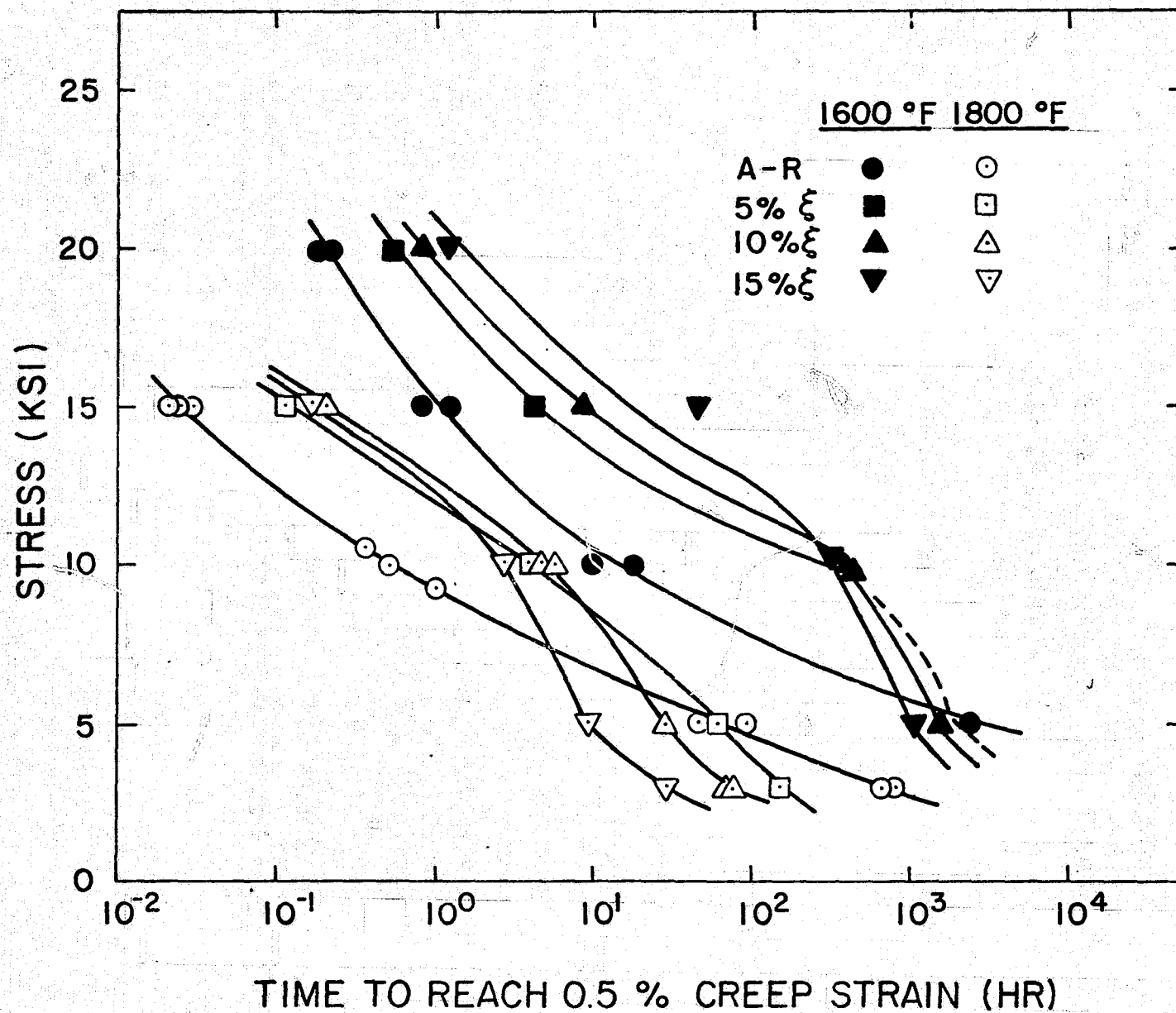


Figure 2.12 The effect of stress on time to reach 0.5% creep strain for Haynes 188 alloy.

At 1800°F, the exposure time for significant improvement is considerably shorter. For one hour exposure, the 15% prestrain provides about 40% increase in design stress; for exposures beyond about ten hours, the thermomechanical treatments tend to shorten the design stress relative to as-received, mill annealed, material. The lower prestrains, however, maintain significant improvement to longer exposures than the higher values. For example, a 5% prestrain still provides a 25% increase in design stress for ten hours exposure at 1800°F.

The reason that the inversion of the effect of thermomechanical treatment shows in Figure 2.12 for both temperatures and only for 1800°F in Figure 2.11 is seen in the creep curves of Figure 2.7. Note that at 1600°F and 5 ksi the minimum creep rate is lowered by a 15% prestrain, and this allows only about 0.3% strain in 600 hours of creep. Apparent substructural instability then sets in and the creep rate increases rapidly so as to cross the as-received curve before 0.5% strain is reached. Note from this figure that the times to reach 0.5% for 5 ksi and 1600°F shown in Figure 2.12 were estimated by extrapolation.

Reference to Figure 2.11 shows that at all conditions of creep at 1600°F and for the as-received condition at 1800°F, the effect of stress on creep rate below about 15 ksi can be represented by the usual power law with a stress exponent of about 5.5. This is about normal for Class II alloys, i.e., those alloys whose stress dependence is not affected by alloying additions (1, 3).

Calculations of the apparent activation energy for creep of as-received material between 1600°F and 1800°F provides a value of 103 kcal/mole. This is significantly higher than the expected value for self-diffusion of about 75 kcal/mole. This is undoubtedly due to lack of substructural equivalence for minimum creep rates at the two temperatures.

As previously mentioned, it is reasonable to expect that the inversion effects noted herein for longer exposure times are related to substructural instabilities. Since these effects are greater for increasing degrees of prior cold work, some form of recrystallization behavior is anticipated. Evidence of grain boundary instability was found using scanning electron microscopy on polished and etched surfaces of specimens which had been creep tested at 1800°F and 5 ksi. Figure 2.13 shows the grain and carbide structure near the fracture zone for an as-received and preoxidized specimen creep tested at 1800°F and 5 ksi. Note the carbides at the grain boundaries and the tendency for some void formation there. Figure 2.14 shows the structure for a specimen prestrained 10%, preoxidized, and then creep tested at 1800°F and 5 ksi. Note that considerable grain boundary migration has occurred, and many carbides no longer reside at grain boundaries. There is also a corresponding decrease in grain boundary void formation, undoubtedly contributing to the increased creep ductility obtained, i.e., 6.5% fracture strain for Figure 2.13 and 13% fracture strain for Figure 2.14.

It has been found possible to correlate the true strain at fracture with a parameter devised from the relation^(1,2):

$$\dot{\epsilon}_{\min} = S \exp (-\Delta H/RT) \left(\frac{\sigma}{E}\right)^n$$

where:

$\dot{\epsilon}_{\min}$ = the minimum creep rate,

σ = the creep stress

E = Young's modulus,

ΔH = the activation energy for creep,

R = the gas constant,

T = the absolute temperature,

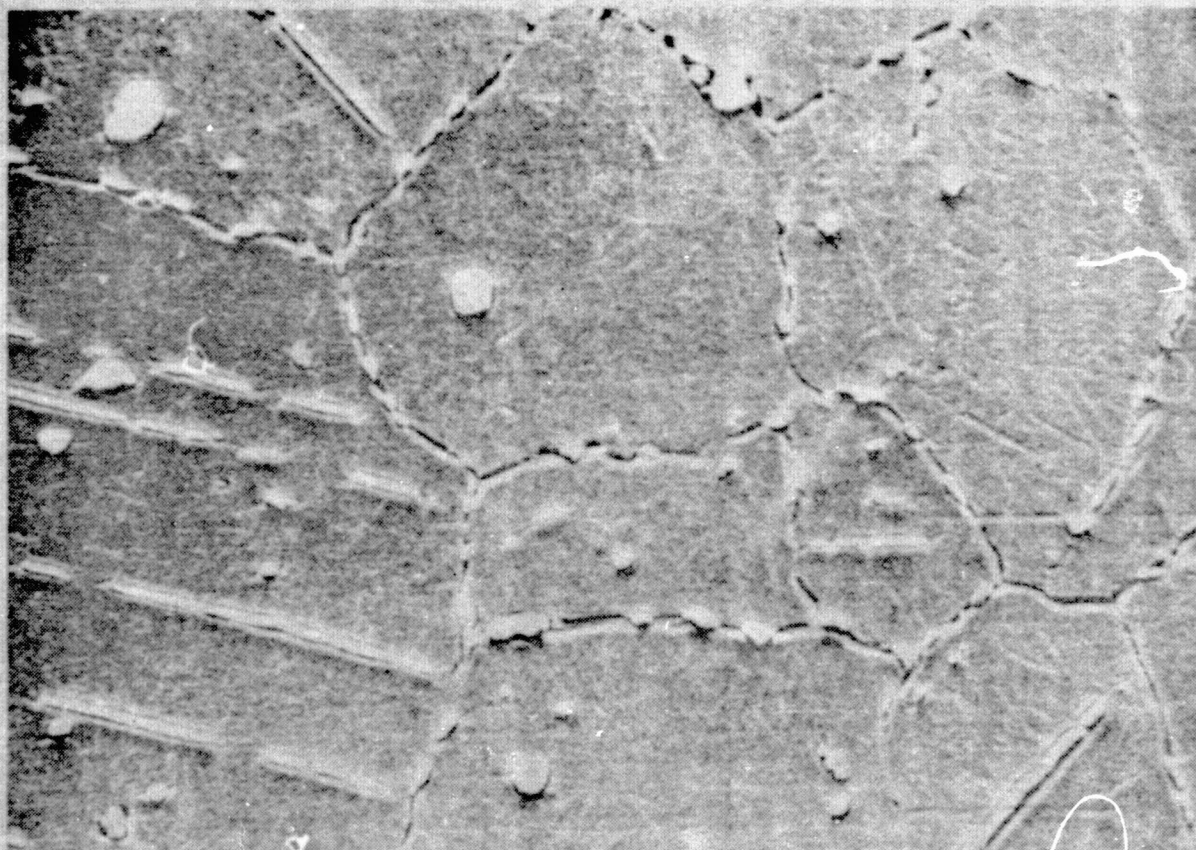


Figure 2.13 Scanning electron micrograph of as-received and preoxidized Haynes 188 alloy after creep at 1800°F and 5 ksi. 1630X.

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Figure 2.14 Scanning electron micrograph of 10% prestrained and preoxidized Haynes 188 alloy after creep at 1800°F and 5 ksi. 1630X.

n = the stress exponent, and

S = a structural parameter.

By noting that S is proportional to $\dot{\epsilon}_{\min}/\sigma^n$ at a given temperature, and that fracture strain is a structural feature, a plot of $\dot{\epsilon}_{\min}/\sigma^5$ vs. true creep strain at fracture was made and is shown in Figure 2.15. The correlation is surprisingly good, considering that fracture strains are plotted for all tests, both as-received and thermomechanically treated. While it is tempting to suggest that lower creep rate provides lower creep fracture strain because of longer exposure times at higher temperatures, no reasonable correlation has been found when fracture life is used in place of minimum creep rate. The correlation suggests that whenever decreases in minimum creep rate are achieved by structural rearrangement in this alloy a corresponding decrease in creep fracture strain will occur. The decrease in fracture strain, however, is no greater than would be obtained when decreases in creep rate are obtained by lowering the stress.

Observation of dislocation substructures by transmission electron microscopy has provided for a number of conclusions regarding the results summarized in Figures 2.11 and 2.12. Typical electron micrographs are shown in Figures 2.16 through 2.25. Discussion of these micrographs is summarized below.

The high degree of solid solution alloying in Haynes 188, combined with its face-centered cubic crystal structure, suggests that the stacking fault energy will be low and that the dislocation arrays formed at low prestrains will be planar in nature.⁽¹⁰⁾ This further suggests that well-defined "cell" structures as found in iron,⁽¹¹⁾ aluminum,^(10,12) and other metals of high stacking fault energy might not be formed. This

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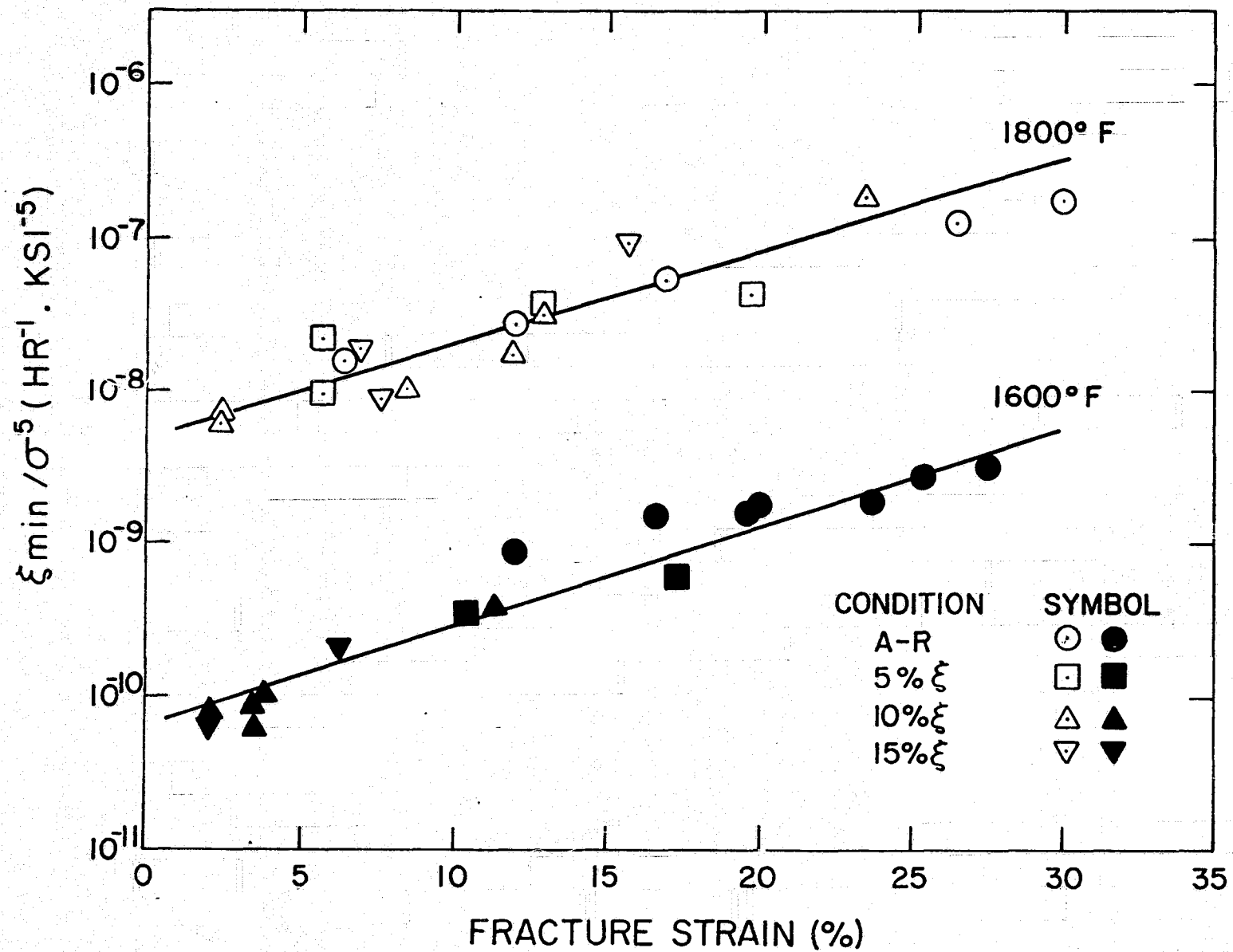


Figure 2.15 The effect of creep rate on fracture strain for Haynes 188 alloy

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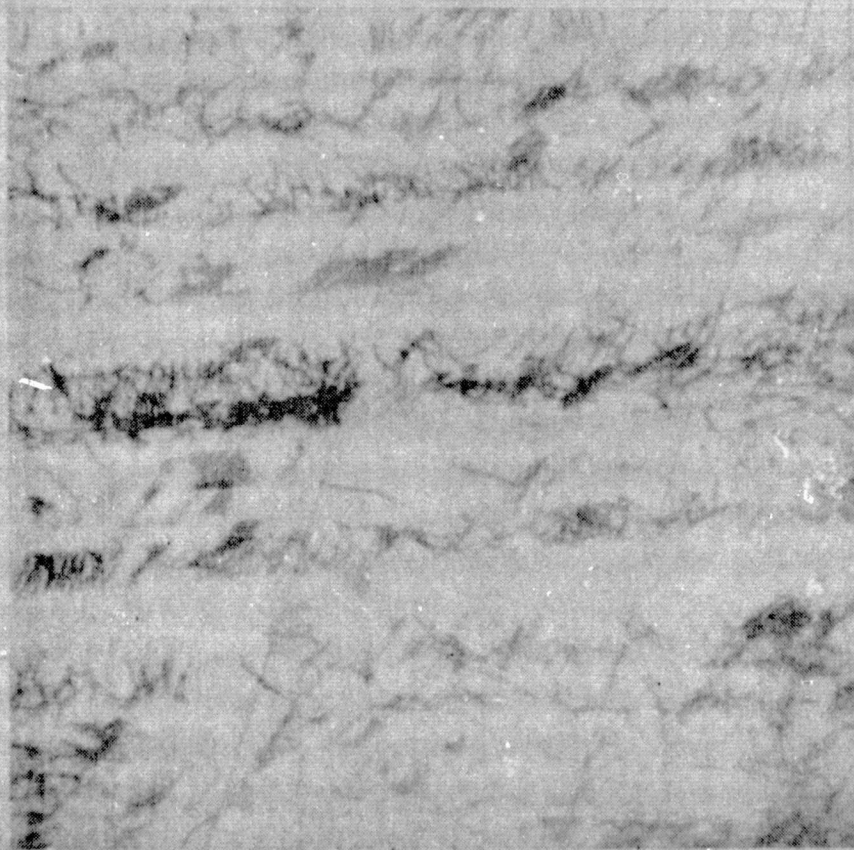


Figure 2.16 Transmission electron micrograph of
as-received, stretch-flattened
Haynes 188 alloy. 32,000X.



Figure 2.17 Transmission electron micrograph of Haynes 188 alloy after 10% prestrain followed by recovery anneal of one hour at 1800°F. 32,000X.

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Figure 2.18 Transmission electron micrograph of as-received and preoxidized Haynes 188 alloy after creep at 1600°F and 15 ksi to the minimum creep rate. 24,000X.

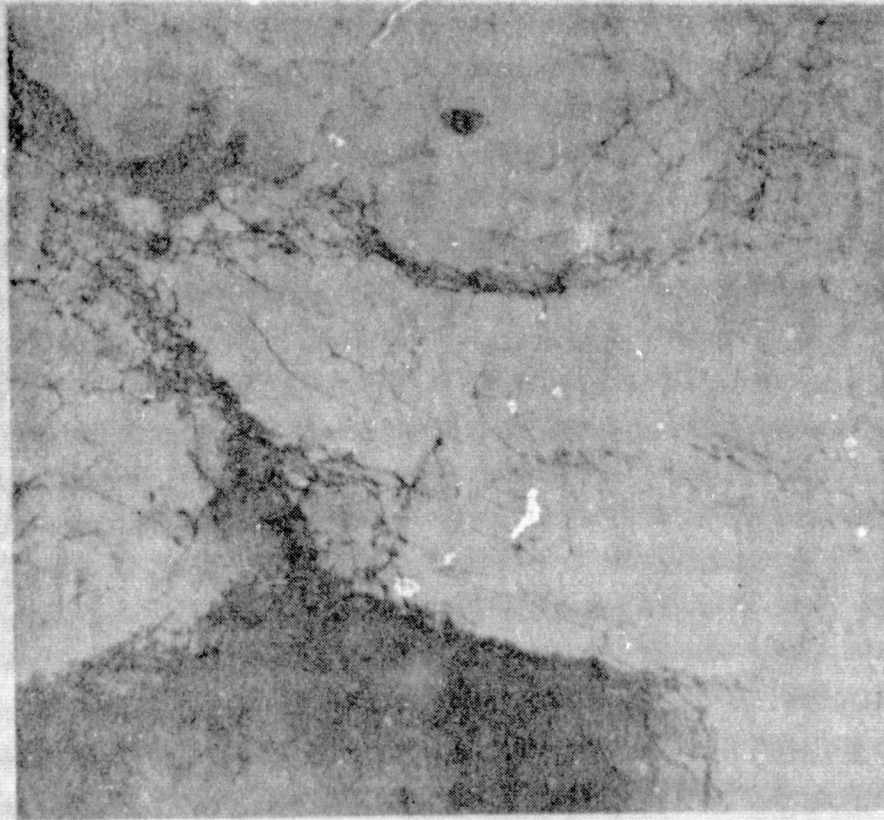


Figure 2.19 Transmission electron micrograph of as-received and preoxidized Haynes 188 alloy after creep at 1600°F and 15 ksi to the minimum creep rate. 28,000X.



Figure 2.20 Transmission electron micrograph of Haynes 188 alloy which was prestrained 10% and then recovered one hour at 1800° F prior to 25 hours creep at 1600° F and 15 ksi. 20,000X.



Figure 2.21 Transmission electron micrograph of Haynes 188 alloy which was prestrained 10% and then recovered one hour at 1800° F prior to 25 hours creep at 1600° F and 15 ksi. 32,000X.

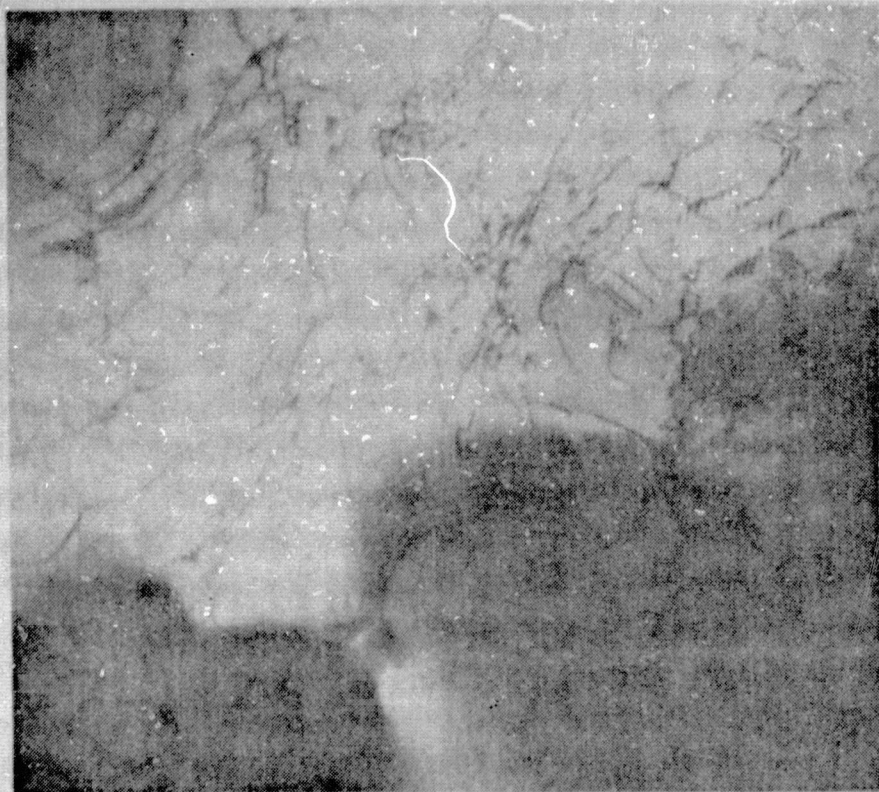


Figure 2.22 Transmission electron micrograph of as-received and preoxidized Haynes 188 alloy after 1005 hours creep at 1800° F and 3 ksi. 32,000X.

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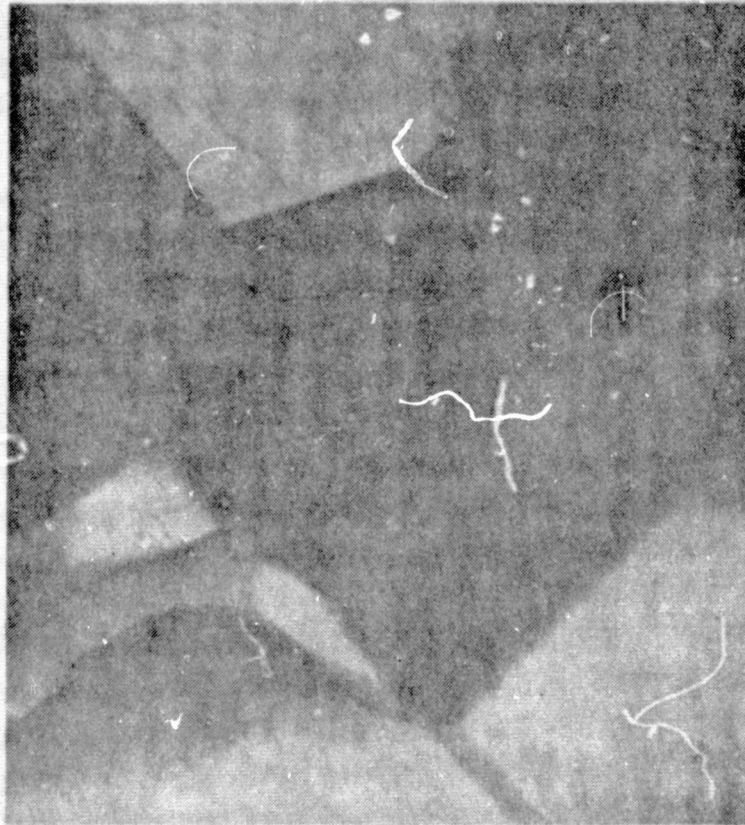


Figure 2.23 Transmission electron micrograph of Haynes 288 alloy which was prestrained 10% and recovered one hour at 1800°F prior to 516 hours creep at 1800°F and 3 ksi. 20,000X.

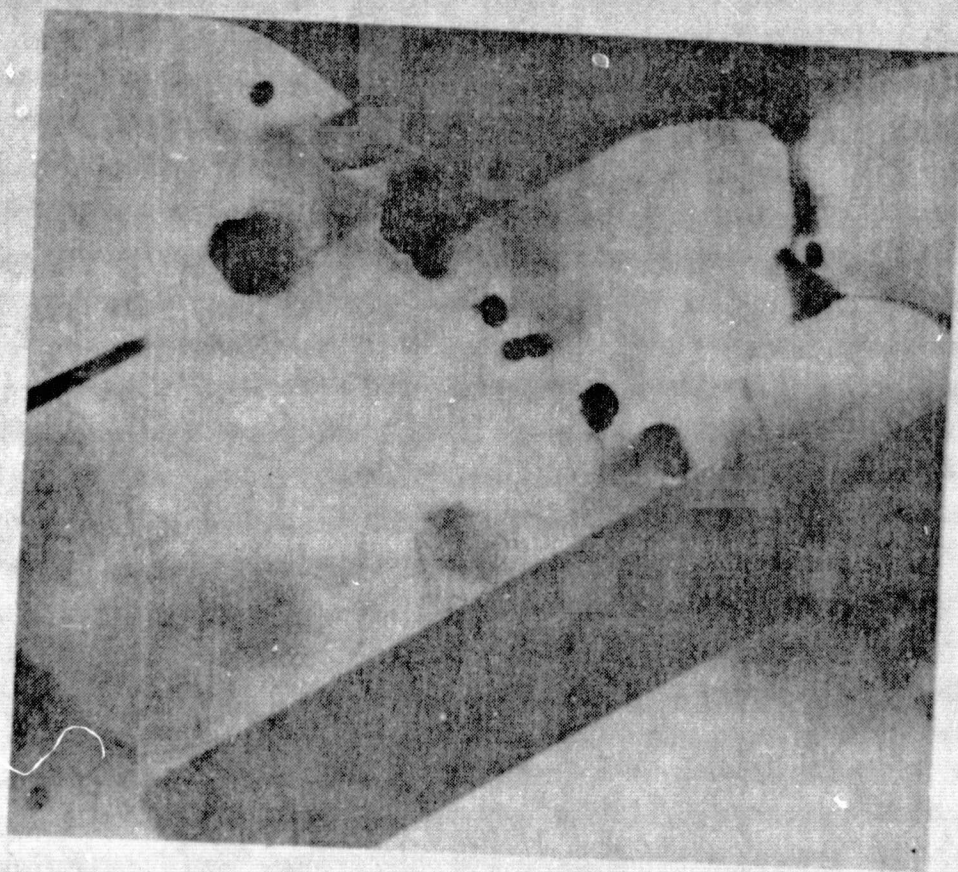


Figure 2.24 Transmission electron micrograph of Haynes 188 alloy which was prestrained 10% and recovered one hour at 1800° F prior to 516 hours creep at 1800° F and 3 ksi. 8,000X.



Figure 2.25 Transmission electron micrograph of as-received and preoxidized Haynes 188 alloy after 25 minutes creep at 1800^oF and 15 ksi. 32,000X.

suggestion is confirmed by the highly planar dislocation arrays found in the stretch flattened as-received sheet as seen in Figures 2.5 and 2.16. The micrograph in Figure 2.17 shows that no clear well-defined subgrains were developed by the 10% prestrain and one hour recovery anneal at 1800°F even though the room temperature flow stress was effectively stabilized. Note, however, that the dislocations in the planar arrays have moved apart and in many cases have reacted with dislocations of other Burgers vectors to provide some degree of network formation.

Typical substructures developed during creep of as-received and pre-oxidized material at 1600°F and 15 ksi to the most creep resistant stage, i.e., the minimum creep rate as identified by "A-R" in Figure 2.8, are shown in Figures 2.18 and 2.19. Figure 2.18 reveals, as expected, that no extensive subgrain formation has occurred at the minimum creep rate for as-received material, although a significant degree of dislocation tangling is evident. These tangles did occasionally appear to provide the beginning of subgrain formation (see Figure 2.19) but the subgrain boundaries were still heavily tangled.

As already noted from Figure 2.11 the minimum creep rate for 15 ksi at 1600°F is about ten times less when 10% prestrain and one hour recovery at 1800°F precedes the creep test. The dislocation substructure which is found at the minimum creep rate for this condition is illustrated in Figures 2.20 and 2.21. Note in Figure 2.20 that many planar arrays appear to be present, but these have become the beginnings of a fine subgrain structure. The misorientation between these regions is evident from the diffraction contrast in Figure 2.20. A higher magnification view of some of the subgrain boundaries is shown in Figure 2.21. The dislocation networks shown represent a clear case

of heterogeneous precipitation on the stacking faults at the extended nodes of highly perfect dislocation networks. The precipitate is presumably $M_{23}C_6$ and formed during the early phases of creep straining due to accelerated kinetics. It should be noted that such heterogeneous precipitation on extended nodes appears to provide the most creep resistant substructure available in Haynes 188, and that this identical conclusion was found during creep in Al - 0.5 At.% Ag by Pawar⁽⁵⁾ when γ' precipitated heterogeneously on the extended nodes of dislocation networks. Examples of the pinned dislocation networks formed in Al - 0.5 At.% Ag have been published by Young and Lytton,⁽⁶⁾ and bear a striking resemblance to those of Figure 2.21.

As previously noted from Figures 2.11 and 2.12, the minimum creep rate was not diminished by prior thermomechanical treatments for creep at 3 ksi and 1800°F; it was increased. In this case, the as-received material had a minimum creep rate which was about 20 times less than for material prestrained 10% and recovered. Reference to Figure 2.4 shows that the $M_{23}C_6$ carbide does not form during static aging until about 600 hours exposure at 1800°F. As seen in Figure 2.22, $M_{23}C_6$ has appeared on some pinned dislocation networks similar to those of Figure 2.21. Note from Figure 2.22 that this region is adjacent to a grain boundary which is still pinned by a precipitate after 1005 hours of creep. When 10% prestrain and recovery precedes creep at 1800°F and 3 ksi, however, the kinetics are accelerated and the grain boundaries do not remain pinned. This is illustrated in Figures 2.23 and 2.24 which shows thermomechanically treated material after 516 hours of creep at 1800°F and 3 ksi. Both twin and grain boundaries are free of precipitates (Figure 2.23) and rows of precipitates are seen without corresponding

boundaries (Figure 2.24). This same situation was shown by the scanning electron micrographs in Figures 2.13 and 2.14. Note from Figure 2.23 that the grains are quite free of dislocation substructure compared to Figure 2.20 where the grain boundaries were stable. It is also significant to note in Figure 2.25 for creep of as-received material at 1800°F and 15 ksi to about the minimum creep rate that tangled subboundaries were able to form adjacent to a grain boundary that was pinned by precipitates. As shown in Figures 2.11 and 2.12, under these conditions, the thermomechanical treatments were effective. Apparently, secondary recrystallization occurs during long exposures due to accelerated precipitate coarsening and the pinned subgrain structure which has been found to be so desirable is unable to form as a stable component of the creep structure. It is clear, therefore, that grain boundary stability is necessary to the beneficial use of thermomechanical treatments for improving the creep behavior of Haynes 188, and that to maintain the required stability at 1800°F requires shorter time conditions, i.e., creep at stresses above about 7 ksi.

SUMMARY AND CONCLUSIONS

Creep tests were performed on Haynes 188 cobalt-base alloy at pressures of 1 Torr and temperatures of 1600°F and 1800°F. Creep stresses ranging from 3 to 20 ksi were employed. The as-received, mill-annealed results were compared to specimens given 5%, 10%, and 15% room temperature prestrains and then annealed one hour at 1800°F to stabilize the dislocation substructures produced. Electron microscopy was used to relate dislocation and precipitate structure to creep behavior. The conclusions reached from this study are as follows:

1. The time to reach 0.5% creep strain at 1600°F increases with increasing prestrain for exposure times of less than 1000 hours; the increase for 15% prestrain is more than a factor of ten. For exposure times of 100 hours, this corresponds to an increase of 60% in the allowable design stress. Above about 1000 hours creep at 1600°F, the beneficial effects of prestrain disappear.

2. Electron microscopy reveals that the highly creep resistant substructure formed consists of dislocation networks where the stacking faults at extended nodes have been pinned by heterogenous precipitation of $M_{23}C_6$ carbides.

3. For creep at 1800°F, the beneficial effects of the thermomechanical treatments used are limited to stresses above about 7 ksi where exposure times are short; below that stress the time to reach 0.5% creep strain is decreased by prior prestrain. Electron microscopy reveals that this is due to enhanced kinetics which allow earlier precipitate coarsening, presumably $M_{23}C_6$, at grain boundaries and subsequent grain boundary

migration. During this migration, dislocation substructure is lost, and the creep rate is correspondingly increased.

4. Grain boundary stability during creep is necessary to the effective use of thermomechanical treatments for improvement of the creep behavior of Haynes 188.

3. THE EFFECT OF DISLOCATION SUBSTRUCTURE ON THE CREEP OF UNALLOYED ALPHA TITANIUM

INTRODUCTION

The results for Haynes 188 alloy given in Section 2 illustrate the improvement in creep behavior which can be obtained in a face-centered cubic alloy for which heterogeneous nucleation on dislocations is possible. It has been demonstrated by Hazlett and Hansen⁽¹³⁾ in nickel and by Chen, Young, and Lytton⁽⁴⁾ in high purity aluminum that prior dislocation substructure in unalloyed fcc metals can favorably alter the course of primary creep. In the latter case, it was demonstrated that formation of a fine prior subgrain structure in aluminum can reduce initial creep rate by at least four orders of magnitude. Such results, however, have not been extended to non-cubic metals, and it was the purpose of this study to determine if similar improvements in primary creep behavior could be obtained in the hexagonal close-packed metal, titanium. It is intended that additional work be performed later on important titanium alloys, e.g. Ti-6Al-4V.

The present creep study begins with the previous work of Ferebee⁽¹⁴⁾ and Pawar⁽¹⁵⁾ who studied the effects of prestrain-anneal treatments on the strength and dislocation structure of unalloyed alpha titanium. They used a 10% room temperature prestrain and various vacuum recovery anneals to determine the fractional flow stress recovery as a function of time. Their flow stress recovery results are summarized in Figure 3.1. Note that effective stabilization of flow stress occurs in 100 hours at 550°C. It was found that a clear subgrain structure was formed by this treatment as shown in Figure 3.2. Pawar⁽¹⁵⁾ found that these subgrains were generally hexagonal in cross-section, the end faces

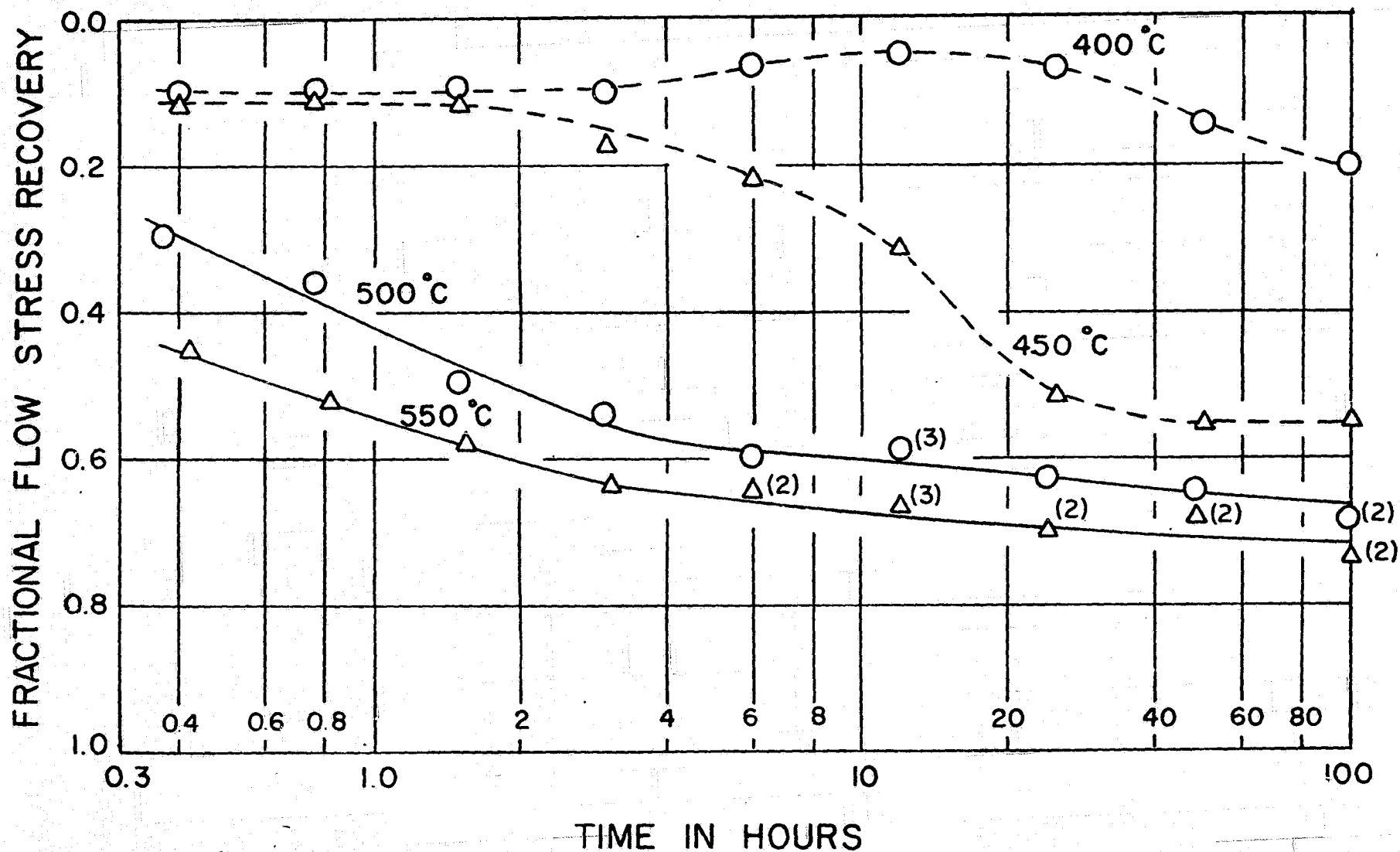


Figure 3.1 Fractional flow stress recovery of alpha titanium as a function of recovery time. The numbers in parentheses indicate the number of tests performed to obtain the average value shown. The dashed curves are from Ferebee (14) and the solid are from Pawar (15).

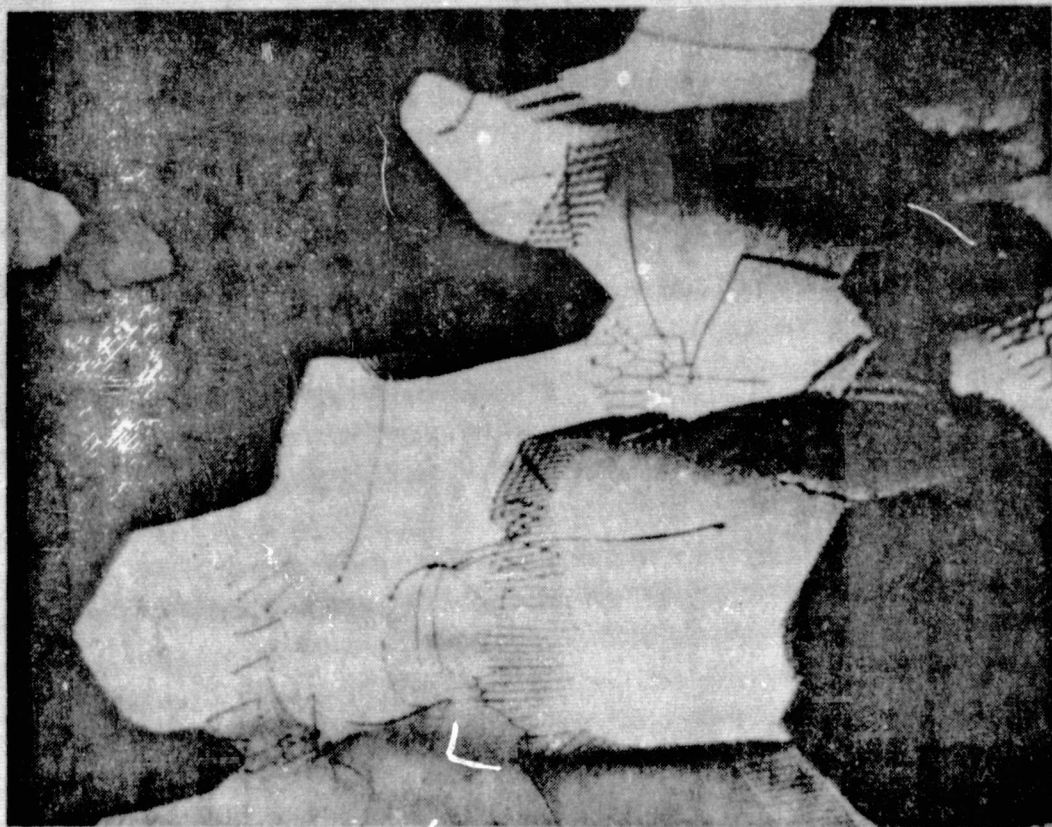


Figure 3.2 Subgrain structure formed in alpha titanium prestrained 10% and recovered 100 hours at 550°C (Foil normal close to $[1\bar{1}01]$), 20,000X.

consisting of hexagonal dislocation networks on $\{0001\}$ planes, and the six planar side faces consisting of parallel glide dislocations lying on $\{11\bar{2}0\}$ planes and in $\langle 0001 \rangle$ directions. The parallel sets of dislocations in Figure 3.2 are glide dislocations lying along $\langle 0001 \rangle$ on $\{11\bar{2}0\}$ planes.

It should also be noted that solution of spot patterns, Kikuchi patterns, and stereographic projections for non-cubic materials is extremely tedious and subject to error. As part of this program, therefore, a previously developed⁽¹⁶⁾ computer program was improved and expanded for computer generation and identification of such patterns for alpha titanium and other crystal structures as well. A paper describing this revised program has been published⁽¹⁷⁾ and a reprint is enclosed herein as Appendix C.

EXPERIMENTAL PROCEDURES

The results presented in this section represent an attempt to determine the effects of prior dislocation substructures like those of Figure 3.2 on the creep of unalloyed alpha titanium at 500°C. Before creep testing could be done, however, a new constant stress creep machine had to be designed and constructed so that the tests could be performed in vacuum. That machine is shown in Figure 3.3. The vacuum annealing furnace constructed for the recovery treatments is shown in Figure 3.4. Thinning apparatus for preparation of thin foils for transmission electron microscopy was also designed and constructed similar to that developed at NASA-Langley (see also Appendix A). The thinning apparatus, patterned after Stieglar and Dubose⁽⁹⁾, is shown in Figure 3.5.

The constant stress creep machine shown in Figure 3.3 is of the parabolic type pioneered by Andrade and Chalmers⁽¹⁸⁾ and has the following characteristics:

1. It has a 5:1 initial lever ratio and will handle specimen loads up to 250 lb.
2. It employs an oil diffusion pump (untrapped) which maintains 6×10^{-5} Torr at the top of the creep chamber during creep tests.
3. It is capable of creep test temperatures up to 1900°F.
4. The furnace can be raised and lowered by motor in about 20 seconds, thereby reducing the waiting time between tests for cooling and heating.
5. The creep machine is designed to handle specimens of 2.0 inches effective gage length. A schematic drawing of the creep specimens used in this study is given in Figure 3.6. The specimen thickness is

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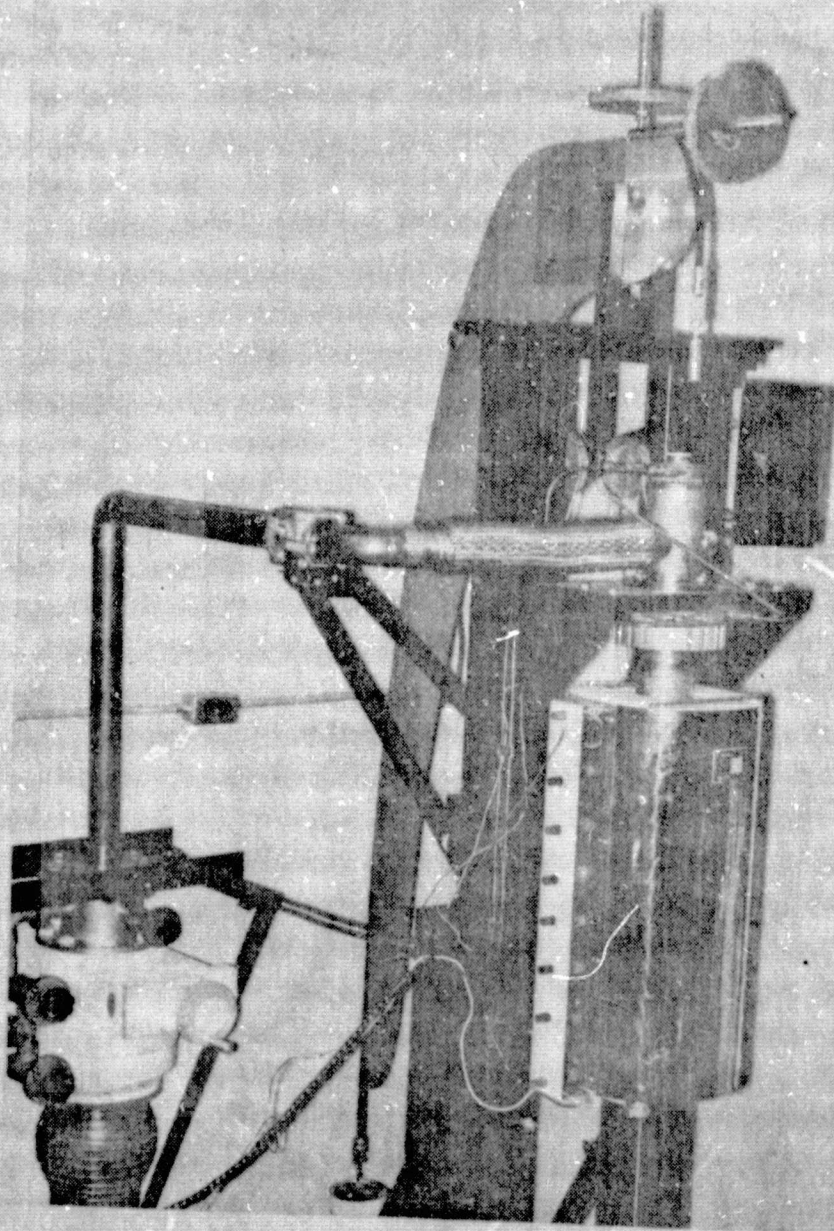


Figure 3.3 Constant stress creep apparatus
for use on titanium.

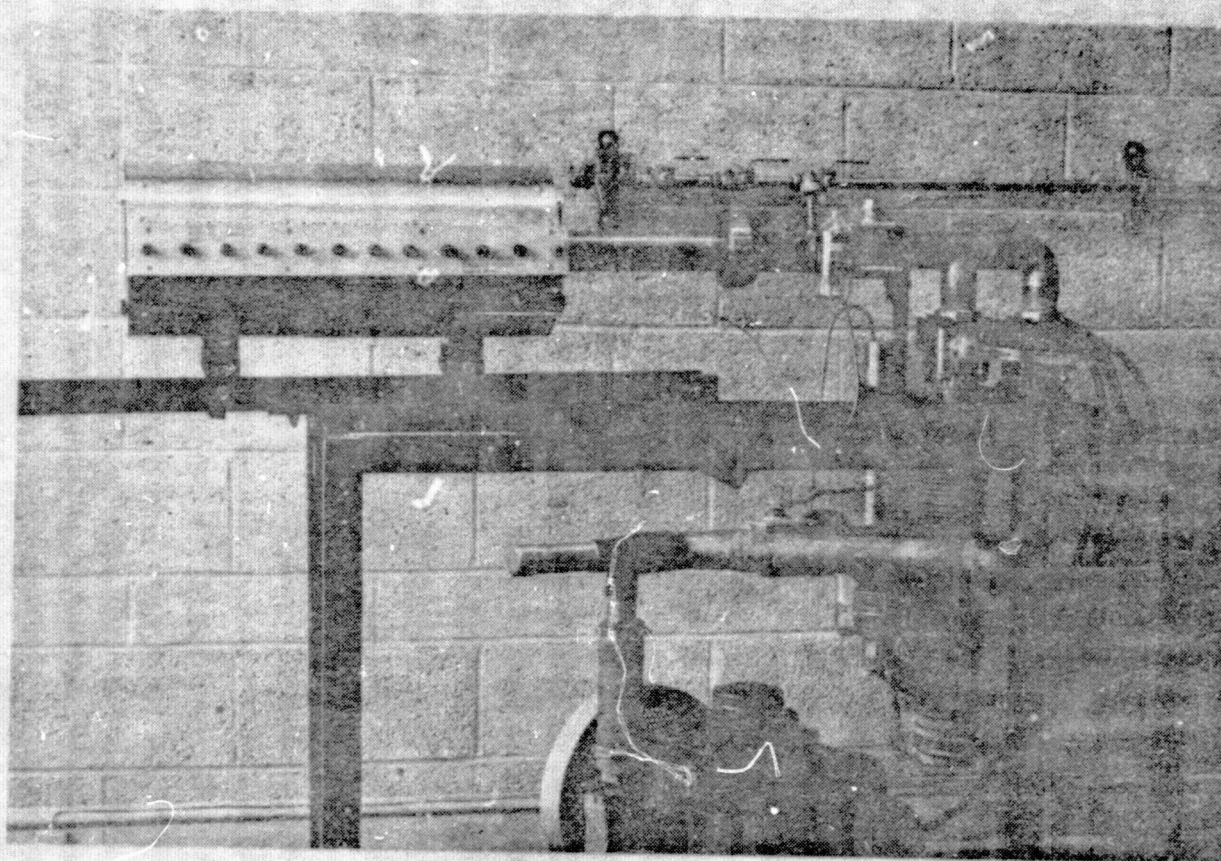


Figure 3.4 High temperature vacuum furnace for use in annealing of titanium.

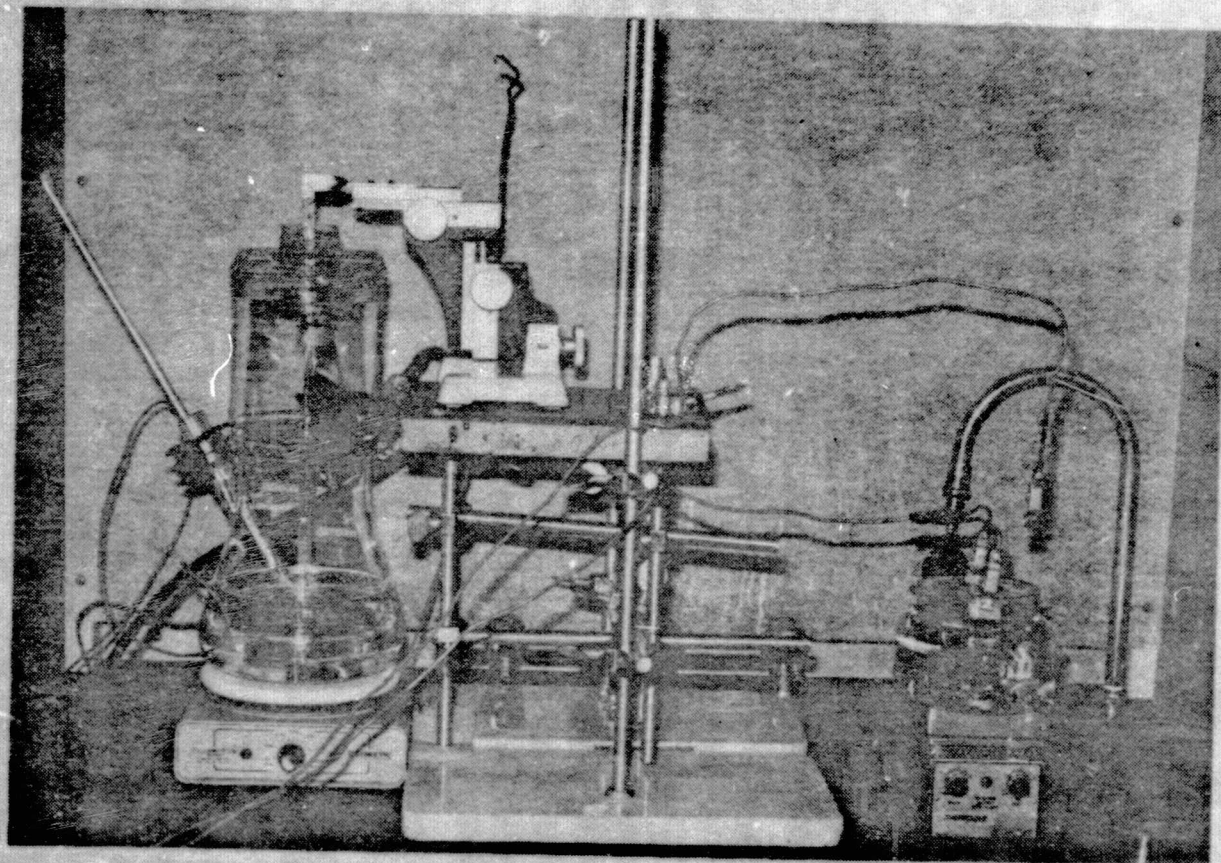


Figure 3.5 Apparatus for preparation of thin foils of titanium for transmission electron microscopy.

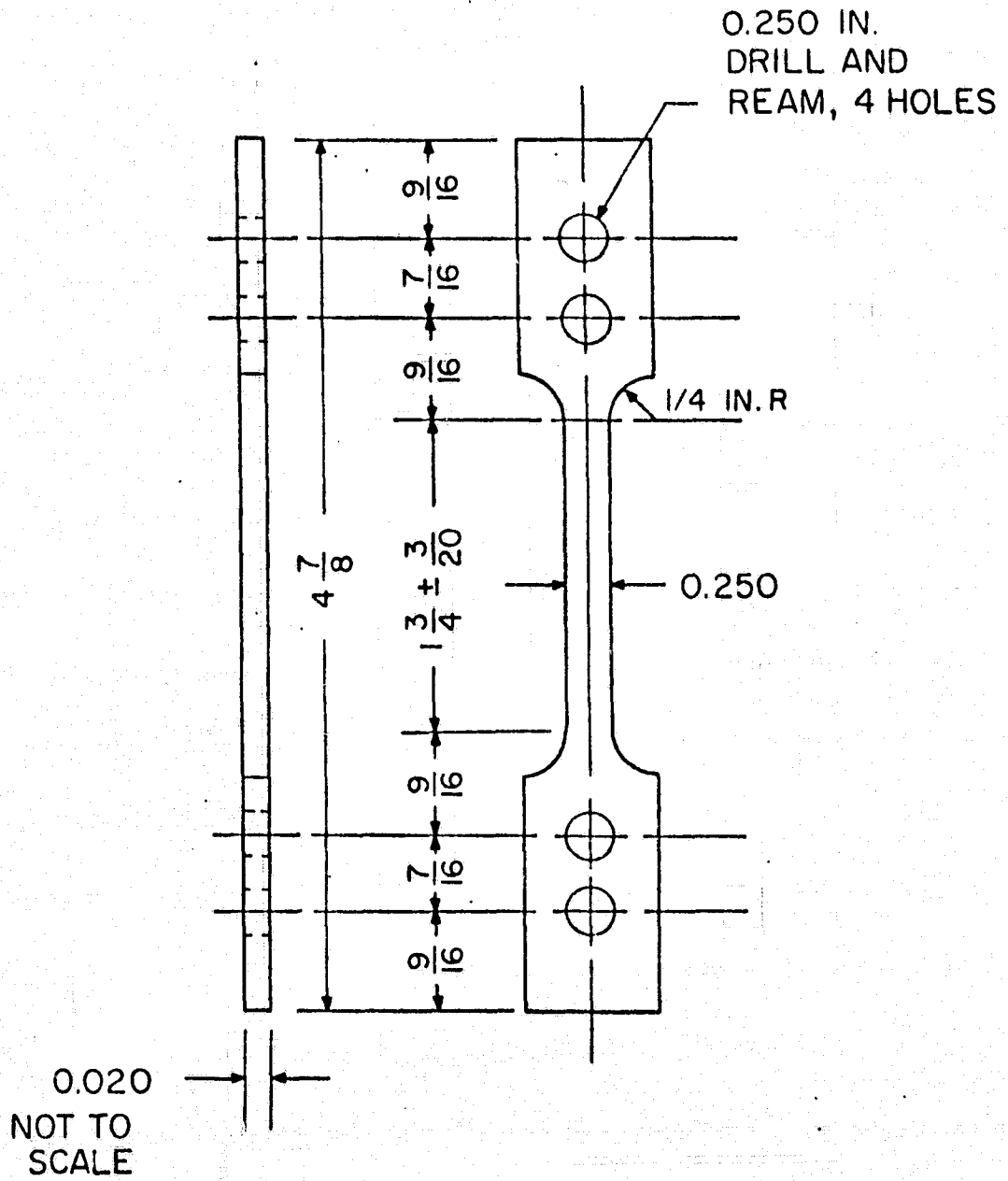


Figure 3.6 Drawing of creep test specimens used for unalloyed alpha titanium.

not an important feature of creep machine design; a thickness of 0.020" was used to minimize thinning while still maintaining several grains across the specimen thickness. Note that the straight section of the specimens is variable. This is because the creep specimen must have 2.0 inches effective gage length after whatever room temperature prestrain is done.

6. Creep strains are measured by LVDT outside the machine at the top of the load train to avoid problems of temperature and outgassing. This necessitates subtraction of the elastic component from total strain, but the high rigidity of the system has kept this correction below 0.002". Creep strains can be measured to the nearest 0.0005 plastic strain with daily variations of excitation voltage limiting the overall strain accuracy to about 0.001.

7. Creep temperatures were attained in a minimum of about 70 minutes by raising the already hot furnace into place. Temperature control was by a solid-state proportioning controller and creep temperature variations were held to about $\pm 2.5^{\circ}\text{C}$.

The material used in this study was Ti-35A sheet of 0.083 inches thickness. Chemical analysis provided with the sheet listed the impurities (weight percent) as: 0.027 C, 0.06 Fe, 0.011 N, 0.004 H, 0.08 O. The long axis of the creep specimens was the mill rolling direction. The as-received sheet was in the annealed condition with a grain size of 0.037 mm. Creep specimens were cold-rolled to 0.020 inch thickness and recrystallized two hours at 1500°F . The resulting grain size was 0.067 mm, giving about eight grains across the specimen thickness.

The preparation of thin foils of titanium was done essentially as described in Appendix A, except for the following:

1. The 2.3 mm diameter discs were cut from the specimens by ultrasonic grinding using a 600 grit silicon carbide slurry and a low-carbon steel tube for a tool. The specimens were first cemented between glass plates for support and the trepanning operation was done through both the glass plates and the creep specimens. No introduction of dislocations was found in annealed samples prepared in this way.

2. The solution used for dimpling consisted of 200 ml methanol, 20 ml H_2SO_4 , and 4 ml HF. The dimpling voltage was 100 volts D.C. and the corresponding current was 200 ma. Dimpling time was about 75 seconds for each side, leaving about 0.002" remaining in the center.

3. Thinning was done at -50 to $-60^{\circ}C$ using a twenty percent solution of perchloric acid in methanol. A thinning voltage of 10 volts D.C. gave perforation in three to five minutes, whereupon the voltage was quickly switched off.

RESULTS AND DISCUSSION

For the purposes of this study, constant stress creep tests were performed at 7250 and 10,000 psi. Most of the creep tests were done at 500°C, but one was done at 527°C and 10,000 psi to provide some information on kinetics. The creep results are summarized in Figures 3.7 and 3.8 which show the creep curves obtained at 500°C for various thermomechanical treatments at 7250 and 10,000 psi respectively. Since the principal interest here was primary creep all tests were terminated after about 10% creep strain.

The following conclusions may be made regarding the creep curves of Figures 3.7 and 3.8:

1. The creep behavior of the as-annealed titanium is not typical of metals and alloys. Following an initial strain, the creep rate diminishes rapidly to a minimum value and thereafter rises toward some new, essentially steady-state value. This is not the "tertiary creep" which signals impending fracture in many metals; it is a gradual shift to an equilibrium substructure as will be demonstrated shortly.
2. The use of either a 10% or a 20% prestrain followed by 100 hours recovery at 550°C did not provide for improved primary creep behavior. For creep at both 7250 and 10,000 psi, the creep rate diminishes only slightly and then establishes the steady-state creep rate before about one percent of creep strain is achieved. Without the prior subgrain structure, 8-10% of creep strain is required before steady-state creep occurs. Note that the use of prior thermomechanical treatments, however, tends to nearly eliminate the initial strain obtained for annealed specimens.

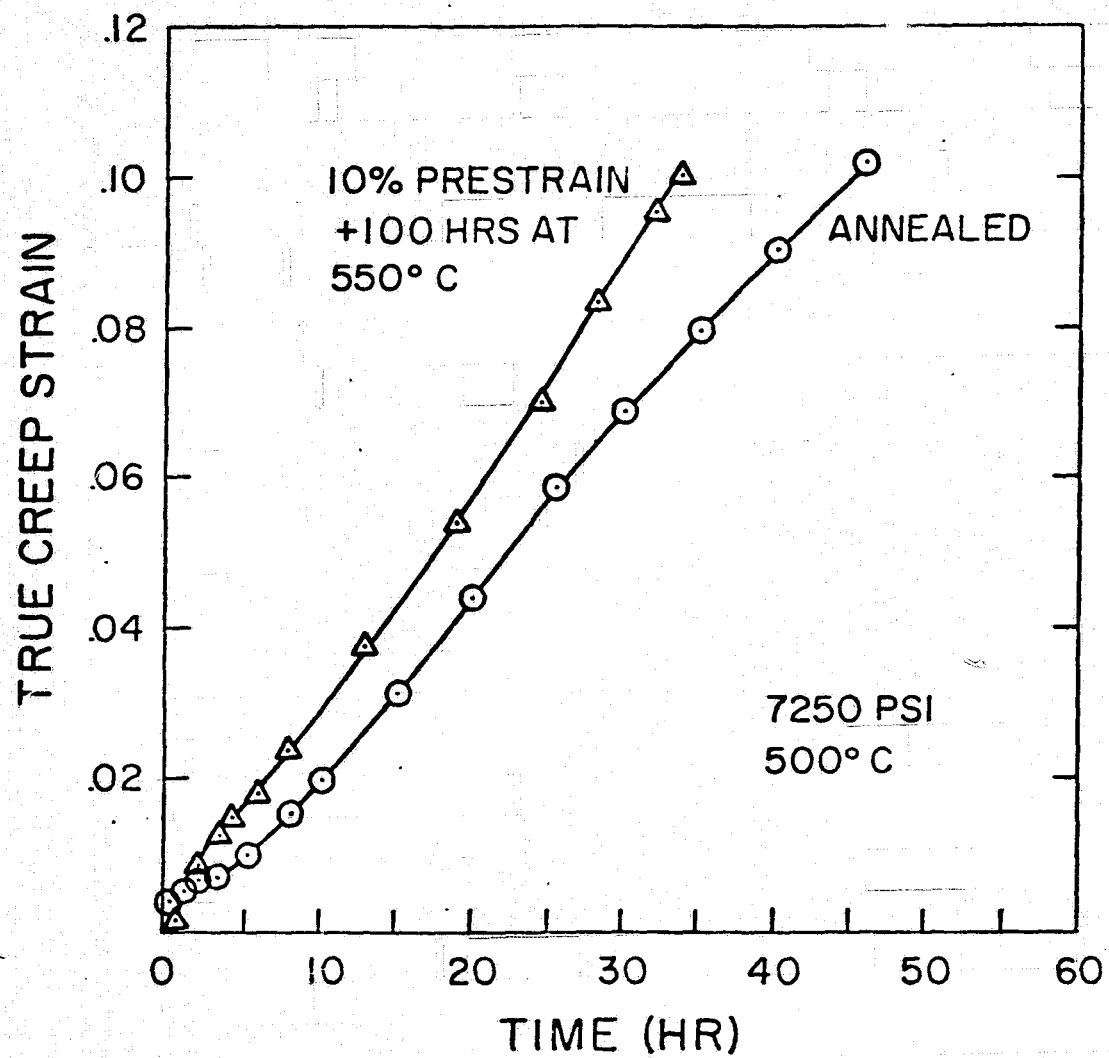


Figure 3.7 Creep curves for unalloyed titanium for 7250 psi and 500°C.

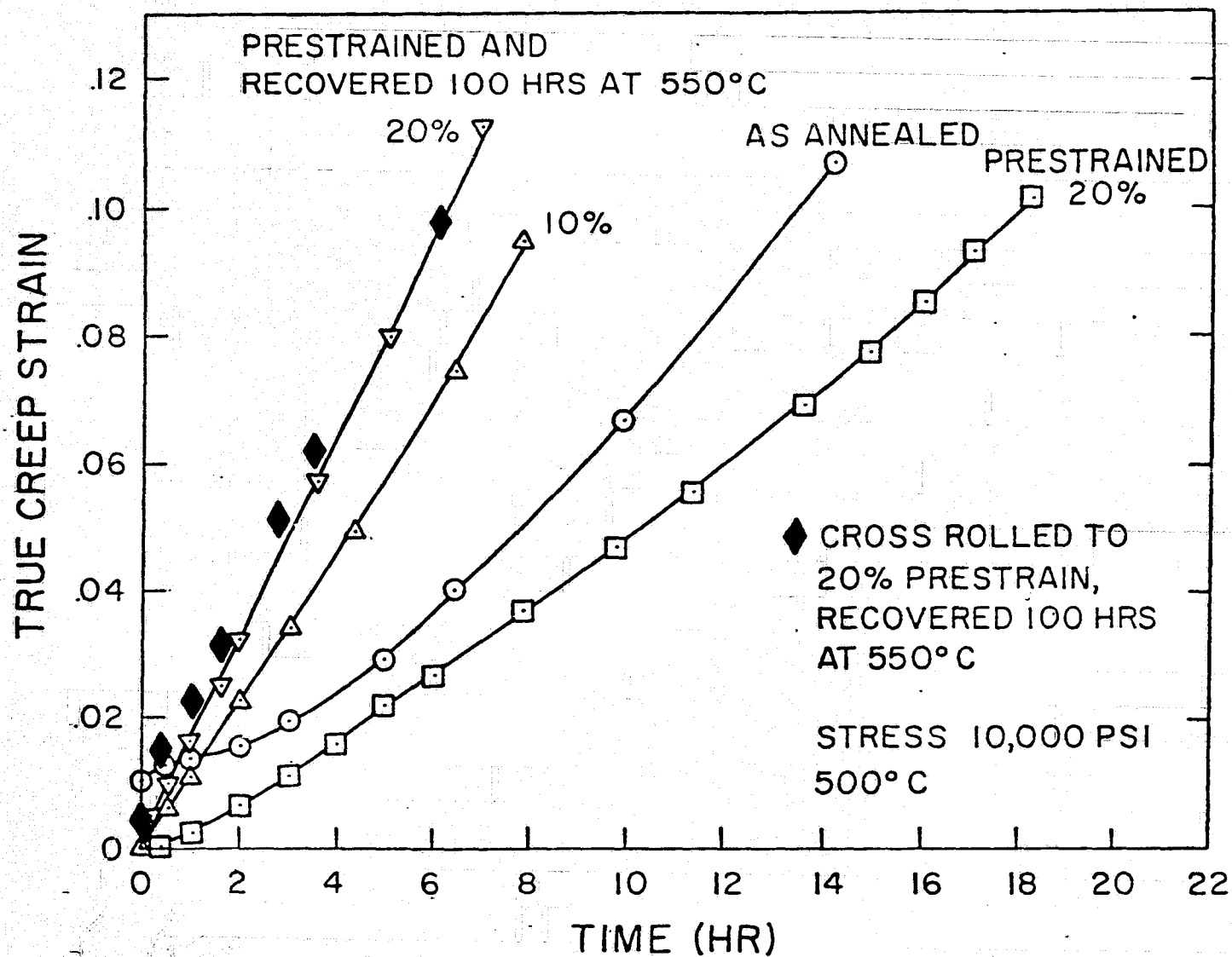


Figure 3.8 Creep curves for various thermomechanical treatments of unalloyed titanium at 10,000 psi and 500°C.

3. It was thought that the observed failure of subgrains to reduce primary creep might be the result of anisotropy induced during prestraining, i.e., a form of "texture weakening". To check on this, a specimen was prepared in the same way as the 20% tensile prestrained specimen, except that the 20% strain was induced by cold cross-rolling 20% prior to 100 hours recovery at 550°C. Note from Figure 3.8 that the creep curve at 10,000 psi was basically independent of the direction of the prestrain employed. It must therefore be concluded that anisotropy induced by the prestrain did not cause the effects observed.

4. The thermomechanical effects observed at the two stresses are essentially similar.

Dislocation substructures were observed after various stages of creep and are shown in Figures 3.9 through 3.14. Figures 3.9, 3.10, and 3.11 represent the structures obtained after 10% creep for three different conditions: (1) as-annealed material crept at 500°C and 10,000 psi (Figure 3.9), (2) as-annealed material crept at 527°C and 10,000 psi (Figure 3.10, and (3) material prestrained 20% and crept at 500°C and 10,000 psi without prior recovery anneal. The important feature to note in all three is that subgrains were formed during 10% creep strain under conditions where they did not exist before the test. In each of these three cases, the creep rate was increasing during subgrain formation, a phenomenon heretofore unrecognized in unalloyed metals. A similar situation has been observed in Al - 2% Mg⁽¹⁹⁾ where it is suspected that mobile dislocation density controls the creep rate.⁽¹⁾ It appears, therefore, that the subgrains formed in titanium as shown in Figure 3.2 provide a ready source of mobile dislocations and that the predominant rate-controlling substructural

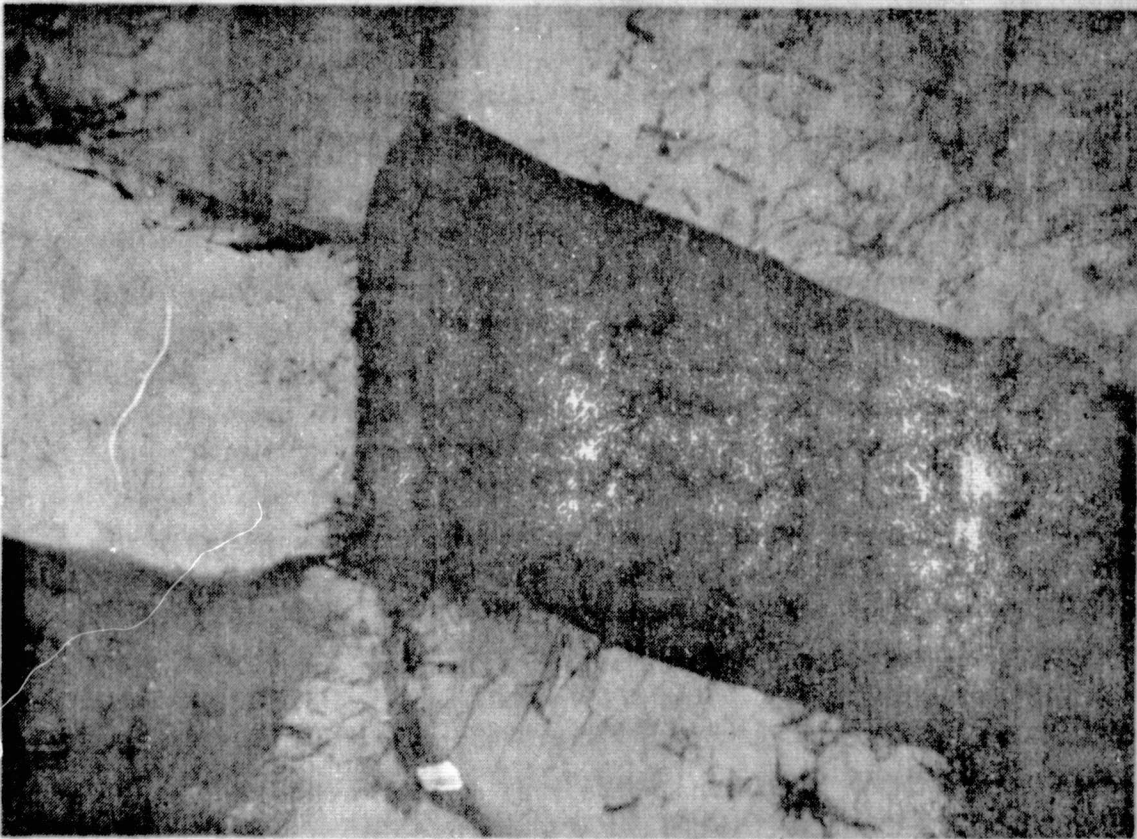


Figure 3.9 Subgrain structure formed in annealed titanium during 10% creep at 500°C and 10,000 psi. 20,000X.



Figure 3.10 Subgrain structure formed during 10% creep of annealed titanium at 527°C and 10,000 psi. 20,000X.

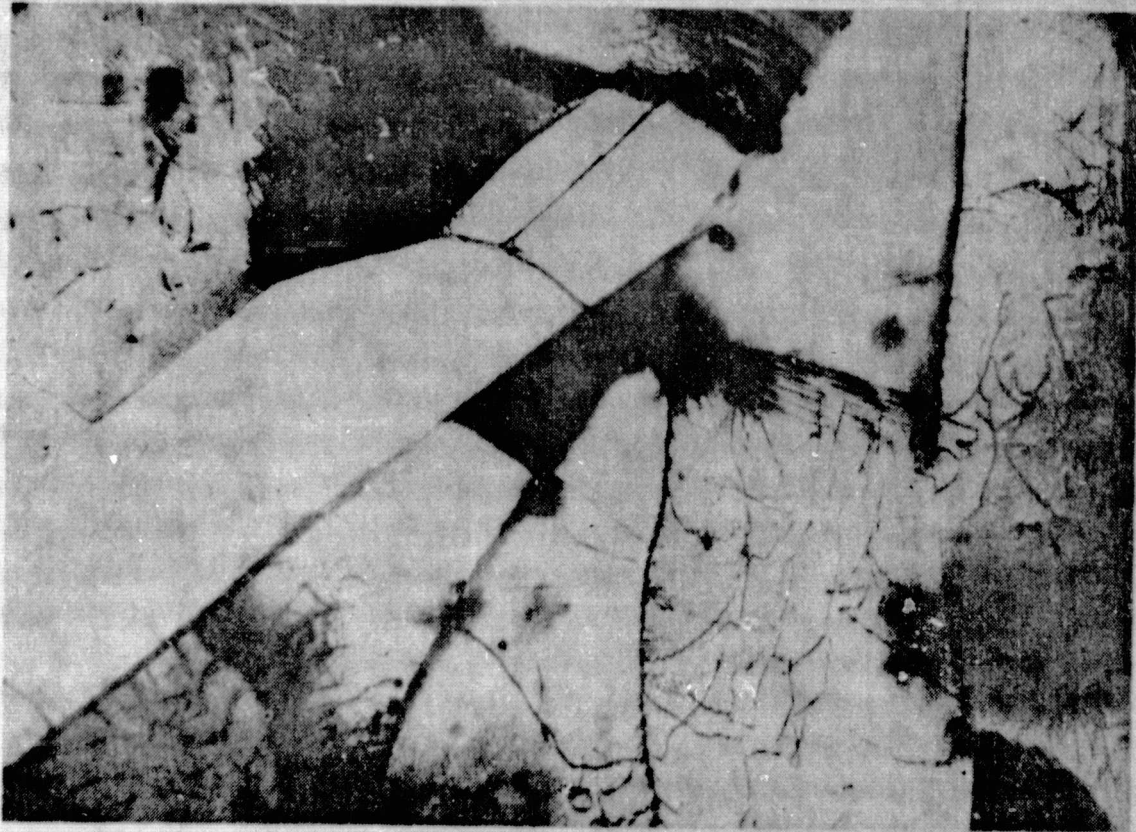


Figure 3.11 Subgrain structure formed during 10% creep of titanium at 500°C and 10,000 psi where 20% prestrain was employed. 20,000X.

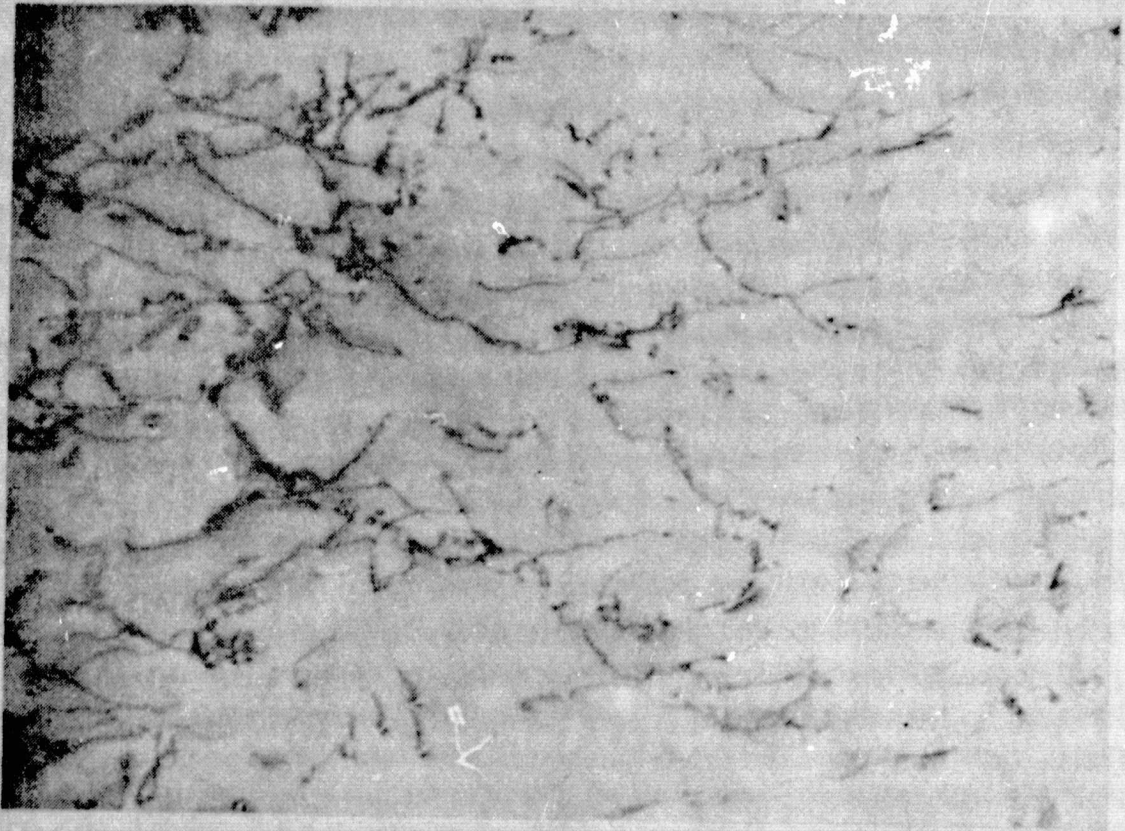


Figure 3.12 Tangled dislocations observed at the minimum creep rate during creep of annealed titanium at 500°C and 10,000 psi. 20,000X.



Figure 3.13 Appearance of prior subgrains in titanium after 0.005 creep strain at 500°C and 10,000 psi. 40,000X.

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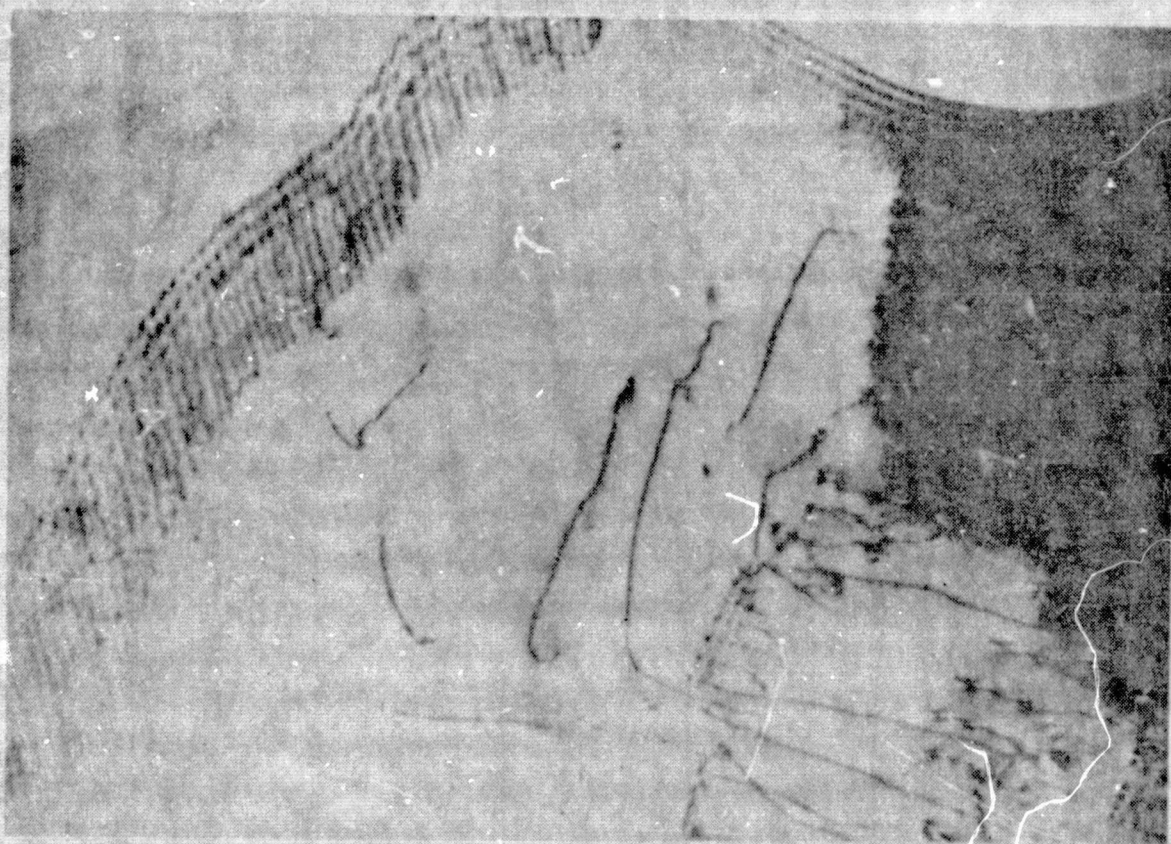


Figure 3.14 Appearance of prior subgrains in titanium
after 0.005 creep strain at 500°C and
10,000 psi. 40,000X.

feature in creep of titanium is the availability of mobile dislocations.

In cases where creep strain occurs by dislocation glide, the creep rate, $\dot{\epsilon}$, would be given by the Taylor-Orowan relation:

$$\dot{\epsilon} = \alpha \rho_m b \bar{v} \quad (3.1)$$

where: α = a geometrical constant,

ρ_m = the mobile dislocation density,

b = the Burgers vector of the moving dislocations, and

$\bar{v}$ = the average glide velocity of the dislocations.

If we assume that the effects of temperature and stress on creep rate are predominantly that of changing the average velocity, we can write:

$$\dot{\epsilon} = \alpha \rho_m b v_o e^{-\Delta H/RT} \left(\frac{\sigma}{E}\right)^n \quad (3.2)$$

where: v_o = a constant,

ΔH = the activation energy for creep,

R = the gas constant,

T = the absolute temperature,

σ = the creep stress

E = the Young's modulus,

n = the stress exponent.

While it cannot generally be safely assumed that the mobile dislocation density is independent of stress and temperature as Equation 3.2 implies, it serves to demonstrate the apparent differences between the effects of prior subgrain structure on creep of aluminum and titanium. In the case of aluminum, dislocation subboundaries consist

of intersecting sets of dislocations of different Burgers vectors, and individual dislocations cannot move quickly and easily from one subboundary to another. The average velocity of a gliding dislocation is therefore decreased because of the time spent in the subboundary regions.

In titanium, Figure 3.2 shows that the typical subgrain walls consist of parallel sets of glide dislocations, and it might be presumed that these are capable of moving easily on their $\{10\bar{1}0\}$ glide planes into the subgrains. It is certainly clear from Figures 3.9-3.11 that dislocations do emanate from the subgrain boundaries eventually formed during creep. Furthermore, Figure 3.8 reveals that when a 20% prestrain is followed by 100 hours recovery at 550°C , presumably creating a finer subgrain size, the creep rate rises even more rapidly with strain towards its eventual steady-state value. This is the opposite of the effect found by Chen, et al.⁽⁴⁾ in aluminum where finer subgrains produced lower creep rates. It is also opposite to the effects of prestrain on creep of nickel as observed by Hazlett and Hansen.⁽¹³⁾

It should be noted that the creep rate for as-annealed material in Figures 3.7 and 3.8 shows an initial decrease to a minimum value, followed by a steady increase as subgrains are formed. It is clear, therefore, that the dislocation structure at this minimum rate is the strongest type available. The dislocation structure found is shown in Figure 3.12. Note that straight dislocation segments are not seen; instead they are helical in nature and quite tangled. Such dislocations could glide only over small segments of their length and in conjunction with considerable dislocation climb. It is believed that these tangles

are generated at the rapid strain rates induced upon initial loading where the initial creep strain occurs in annealed material. They provide a minimum creep rate by keeping both the average velocity and mobile dislocation density to low levels. Gradually, however, they straighten with time and begin to glide and interact to form the subgrains as seen in Figures 3.9 to 3.11.

To determine if the subboundary dislocations generated by the 10% prestrain and recovery anneal for 100 hours at 550°C are mobile, a creep test was performed on suitably pretreated material at 500°C and 10,000 psi and stopped at only 0.005 creep strain. Transmission electron micrographs of typical subgrain boundaries are shown in Figures 3.13 and 3.14. Note in each case that glide dislocations do move readily out of the subboundaries and that tangling is not evident. This presents clear proof that subgrain boundaries in titanium do provide a ready source of glide dislocations during creep and therefore do not represent a means of enhancing creep resistance unless the dislocations can be effectively pinned; e.g. by heterogeneous precipitation as demonstrated for Haynes 188 alloy in Section 2.

The foregoing arguments suggest that the tangled dislocation structure which forms during prestraining^(14,15) might exhibit improved creep resistance over as-annealed material. This prestraining provides a ragged "cell" structure, similar to a subgrain structure, but with low dislocation mobility due to heavy tangling. Figure 3.8 also shows a creep curve for a 20% prestrain without prior recovery treatment except for that which occurred during the 70 minutes required to go from room temperature to 500°C. Note that this curve combines a low initial strain with the longest times to given creep strain levels, e.g., the time to reach 1% creep strain is about three hours compared to less than 30 minutes for material containing subgrains.

Although the present study did not include the range of testing needed to clearly establish the activation energies and stress laws for creep, some observations can be made. Since a clear indication of substructural equivalence, namely the formation of subgrains, was only found after about 10% creep strain, it may be concluded that the true steady-state rate for 10,000 psi at 500°C is that towards which all the curves of Figure 3.8 are approaching. This rate is about 0.017 per hour. It is about 0.0034 per hour for 7250 psi at 500°C, and about 0.032 per hour for 10,000 psi at 527°C. Applying these numbers to Equation 3.2, and assuming structural equivalence, provides an activation energy for creep of 28,800 calories per mole and a stress power law exponent of five. The stress exponent is in reasonable agreement with the previous results of Kehoe and Broomfield⁽²⁰⁾ for unalloyed titanium. Their values ranged from 4.5 to 6.8 with decreasing stress; their value of 4.5 was obtained at about the range of stresses used here. These authors, however, also obtained activation energies for creep ranging from 69 to 72 kcal per mole, more than double the value obtained here. The difference is undoubtedly due to the use of minimum creep rate by Kehoe and Broomfield, a situation not likely to produce meaningful results in view of the substructural results reported herein. It is interesting to note that the activation energies for self-diffusion reported by Dymant and Libanati⁽²¹⁾ for Ti and Zr are 35.9 and 27.0 kcal per mole, respectively. Zirconium is believed by many investigators to behave similarly to alpha titanium, and the creep of zirconium has been studied by Ardell and Sherby.⁽²²⁾ These authors found that the activation energy for creep of zirconium increased with decreasing stress. Tests performed recently on titanium have suggested that creep at 500°C and 5000 psi also produces higher activation energies.⁽²³⁾

SUMMARY AND CONCLUSIONS

The effect of various thermomechanical treatments on the constant stress creep of alpha titanium has been studied at 500°C for stresses of 7250 and 10,000 psi. Creep behavior was correlated with dislocation structure as observed by transmission electron microscopy. The conclusions made from this study are as follows:

1. The predominant rate-controlling structural feature during creep of alpha titanium is the availability of mobile dislocations.
2. The use of thermomechanical treatments to produce prior subgrain structure in titanium decreases creep resistance because the parallel glide dislocations in the $\{11\bar{2}0\}$ subgrain walls move readily under stress.
3. The creep rate in titanium increases as subgrains form during creep and increases with finer subgrain size.
4. The best creep resistance in titanium is obtained by prestraining to produce a tangled cell structure and applying the creep stress before recovery can occur; this provides both a low average velocity of dislocations and a low density of mobile dislocations.
5. The stress power law exponent obtained between 7250 and 10,000 psi was 5 and the activation energy for creep was 28,800 calories per mole.

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APPENDIX A

Procedures for Thinning
of Foils of Haynes 188 for
Transmission Electron Microscopy

OPERATION OF THINNING APPARATUS

(Numbers in parentheses refer to Figure A1)

1. Prepare disc specimens of 3 mm in diameter and about 1 mm in thickness from bulk specimens (preferably using electron discharge machine). Grind off surface oxide, if any.
2. Prepare polishing solution, and replace or dilute dimpling solution, if necessary.
3. Fill up liquid N_2 in the container (1), and fix the pipe line.
4. Open the liquid N_2 main valve (2), the N_2 gas inlet valve (4), and the two control valves (6) and (7).
5. Open the main valve on N_2 gas tank (5), and regulate gas pressure at 2-3 psi as read on the pressure gauge (3). (This will increase the liquid N_2 flow through the cooling coils. When rapid cooling is not needed, close the main valve on gas tank to conserve both liquid N_2 and N_2 gas.)
6. Turn on the main switch on switch box A (SA), with the other five on-off switches on this box turned off.
7. Make sure all switches on the two DC power supply units (21) and (30), the light source unit (33), and the electrolytic polishing controller (32) are OFF. Turn on the main switch on switch box B (SB). (The five on-off switches on this box should be ON all the time.)

Dimpling Procedures

8. Close the control valve (6) when temperature as read on the thermometer (11) has dropped to a desired point. If the solution is under-cooled, bring the solution to the upper compartment of dimpling jig (close air outlet valve (14), open air inlet valve (13), and turn on air pump (12)), and heat the solution with heating tape (23).
9. Turn the selector switch on the DC power supply unit (Nabatron 610B) (21) from "OFF" to "DC-OFF". Wait for 2 minutes, and press the "HV-START" button.
10. Turn on illuminating light (20).
11. Close the air outlet valve (14), and turn on air pump (12).
12. Adjust air inlet valve (13) to initiate jet action, and then maintain air pressure at 40-50 mm Hg as read on the mercury gauge (15) (take about 1 minute to obtain stabilized pressure).

13. Hold the disc specimen (17) at the edge of specimen with the platinum-tipped tweezer (18). Attach the tweezer to the micromanipulator (19).
14. Lower the tweezer, and adjust the specimen's position so that the cathode-to-specimen distance is about 1 cm, and that the jet is directed toward the center of specimen.
15. Turn the selector switch from "DC-OFF" to "+600" to start dimpling action, and immediately adjust the DC voltage using COARSE and FINE knobs to obtain the desired DC current (tap the ampere meter while reading). (The DC voltage should be kept below 300 volts. If the voltage with the desired current is over 300 volts, stop the dimpling process immediately. The voltage can be reduced by decreasing the cathode-to-specimen distance.)
16. Turn the switch from "+600" to "DC-OFF" when dimpling time is reached, and immediately remove the specimen away from the jet.
17. Turn off air pump, and open air outlet valve to allow solution to drain back to the lower compartment.
18. Examine the dimple shape. Flat-bottomed dimple is desirable.
19. Turn the disc specimen front-side-back, and repeat the dimpling process (Steps 11 to 18).
20. Repeat Steps 11 to 19 for other disc specimens.
21. Turn the selector switch to "OFF". Turn off illuminating light.

Polishing Procedures

22. Make sure that the inlet and outlet ends of the cooling system for polishing are properly located. Turn on the tubing pump (25). (DON'T reverse the pumping direction without first stopping the motor.)
23. Turn on the stirrer (26).
24. Set the micromanipulator above the polishing dish.
25. Turn on BLOWER and LIGHT on the light source unit, when polishing solution has been cooled to the desired temperature.
26. Lower the tweezer, and adjust the specimen position so that the intense light transmitted through the fiber optics (29) is focused at the center of specimen, and that the polishing solution circulates tangentially to the dimpled surfaces of disc specimen.
27. Set RANGE on the polishing controller at "3", ZERO dial at "500", and the red microampere needle at "6".

28. Turn on POWER switch, and set POLISH switch at "RESET".
29. Adjust ZERO dial so that the black needle on the microampere meter barely starts to move at ranges below "0".
30. Set POLISH switch to "AUTO."
31. Turn on POWER switch on the DC power supply unit (DCR80-10A) (30) and immediately set the DC voltage at the desired point.
32. Adjust ZERO dial, if necessary, during the course of polishing to keep the black needle at slightly below "0".
33. Turn off the DC power switch periodically at about 10-minute intervals. Remove specimen from tweezer. Change the point of contact between specimen and tweezer to prevent preferential polishing around point of contact occurring at the same location on the disc specimen.
34. Remove specimen from polishing solution when perforation occurs. When the dimpled disc is perforated, light passing through the hole or holes will be transferred into electric signals, which cause a quick swing of the black needle to pass the red needle, and shut off DC current.
35. Rinse the specimen carefully (dipping up and down slowly) in 3 cleaning baths with ethyl alcohol.
36. Dry the specimen gently in stream of hot air, with specimen located about 3 feet away from the drier and oriented tangentially to the air flow. The specimen is ready for TEM examination.
37. Repeat Steps 26 to 36 for other dimpled specimens.
38. Turn off DC power switch, stirrer, tubing pump, and the main switches on the two switch boxes.
39. Empty the polishing solution.
40. Loosen the hose clamp which tightens the rubber hose to the mouth of liquid N₂ container.

SOLUTIONS FOR THINNING OF HAYNES 188

The operating conditions described below are derived from experience on H188 cobalt-base alloy.

Solutions

The dimpling solution (1000-1200 cc) contains 33% conc. HNO_3 and 67% methyl alcohol by volume. It can be repeatedly used for dimpling of 60-80 specimens.

The polishing solution (200 cc) contains 10% HClO_4 and 90% ethyl alcohol by volume. Fresh solution is preferable. It can be used for polishing of 6-8 specimens at a time.

Thinning Conditions

Variables	Dimpling		Polishing
	<u>Solution Annealed or Short-Time Aged</u>	<u>Long-Time Aged</u>	
Solution Temperature	0-5°C	10-15°C	0- -10°C
Cathode-Specimen Distance	~1 cm	~1 cm	3-4 cm
DC Current	200-240 ma	240-280 ma	---
DC Voltage	<300 volts	<300 volts	20-22 volts
Thinning Time*	120 sec. @ 200 ma 100 sec. @ 240 ma	100 sec. @ 240 ma 85 sec. @ 280 ma	Varies (10 min-1 hr)

*The dimpling time listed is per side, and is applied to discs started with ~0.023 inch in thickness. The dimpling rate is about 0.005 in/min at 200 ma. A dimpled specimen with ~0.002 inch in thickness at dimpled flat is desirable. The polishing time depends heavily on the dimpling time elapses. One additional second added to the total dimpling time will reduce the polishing time by approximately one minute.

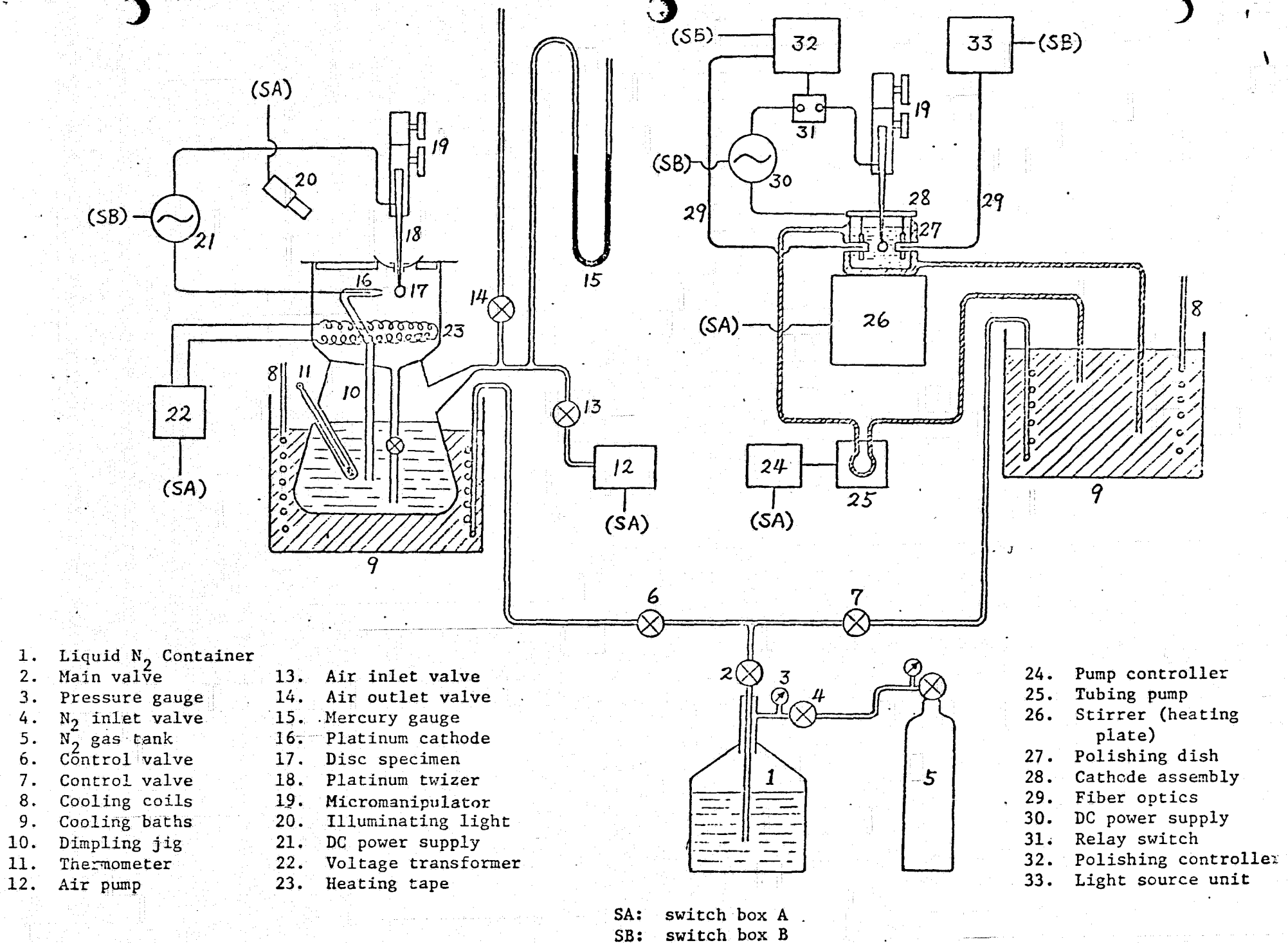


Figure A1. Schematic View of Two-Stage Thinning Apparatus.

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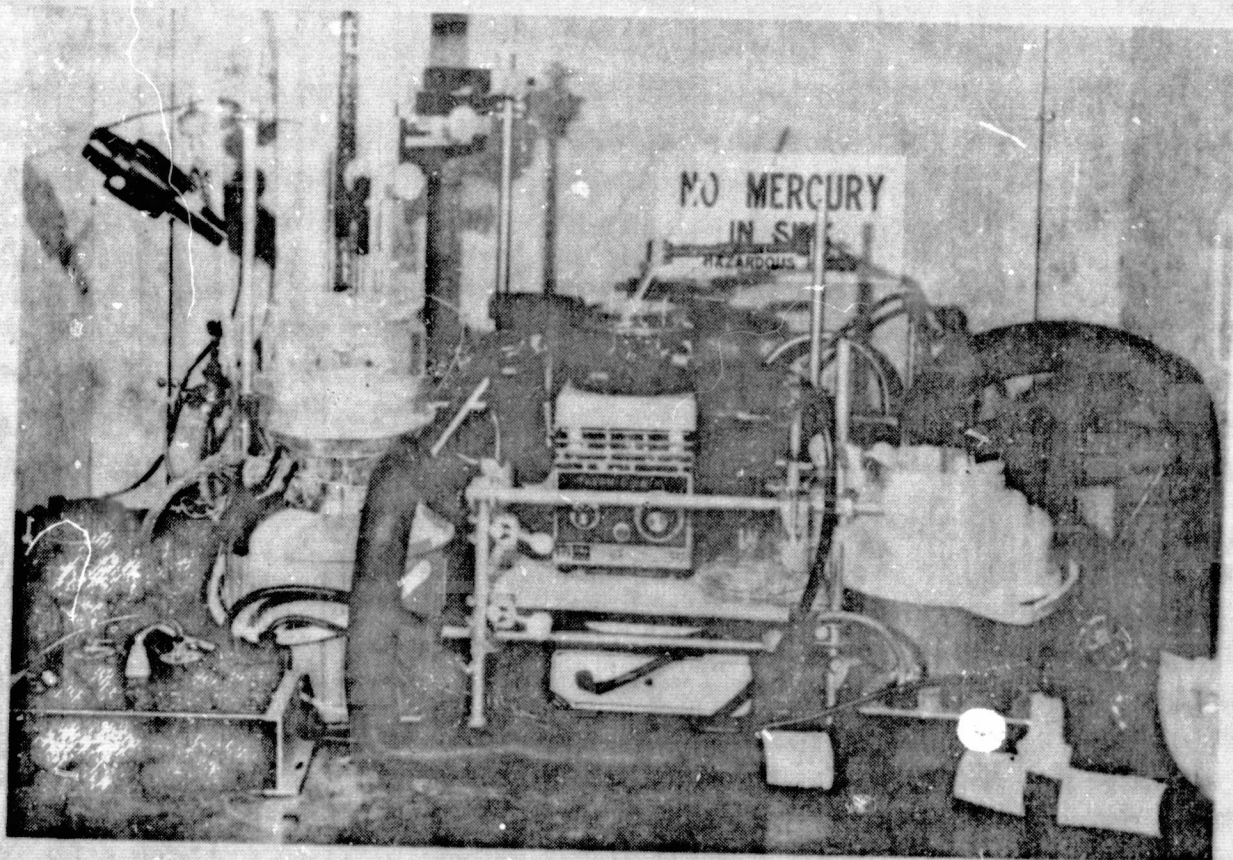


Figure A2. Two-stage thinning apparatus as assembled at NASA-Langley Research Center.

Appendix B

The Accuracy of Simple Beam Axis Solutions
from Kikuchi Patterns

(Manuscript submitted to
Transactions ASM-AIME)

by

C. T. Young

and

J. L. Lytton

THE ACCURACY OF SIMPLE BEAM AXIS SOLUTIONS FROM KIKUCHI PATTERNS

ABSTRACT

Five simple beam axis solutions from Kikuchi patterns, i.e., the 3-pole, the 3-normal, and approximate 3-normal, the 2-pole/1-normal, and the 1-pole/2-normal solutions, are described. The accuracy of each solution was evaluated by determining the angular divergence of ten or more beam axes obtained from the same pattern. The results indicate that the 3-normal and the approximate 3-normal solutions are the most accurate. Beam axes so determined are accurate to better than 0.07° , and are virtually independent of uncertainties in the camera length. For the other solutions in which at least one Kikuchi pole is used to formulate the equations, the beam axis may be determined to 0.14° if the camera length is obtained from Kikuchi pole separation, and to 0.7° if the camera length is computed from Kikuchi pair separation.

INTRODUCTION

Kikuchi patterns have been used in transmission electron microscopy for accurate crystal orientation determination⁽¹⁻¹⁰⁾, bicrystal misorientation calculation⁽¹⁰⁻¹³⁾, and other crystallographic analyses^(2, 14-17). The three-pole solution^(1,2,9,10) has been the simplest and most generally used technique in solving for the electron beam axis of a Kikuchi pattern; however, the accuracy of this solution has not been examined quantitatively. The accuracy of several other methods has been evaluated. The orientation obtained from the one-pole solution of Otte et. al.⁽³⁾, which employed a rather complicated calculation and required a second Kikuchi pole for unique solution was reported to be accurate to $\pm \frac{1}{2}^\circ$. The approach given by Sheinin and Cann⁽⁴⁾ was claimed to be accurate to $\pm 0.05^\circ$, but it required

two exact Bragg reflections and was limited in its application. A simple approximate method of Ryder and Pitsch⁽⁵⁾ was found to be accurate within $\pm 0.1^\circ$ ⁽⁶⁾. In this approach, strong diffraction spots from more than one zone together with their corresponding Kikuchi pairs were used, and values of lattice parameter, electron wavelength, and effective camera length were not required in the calculation. A similar, but exact, method which involved a more complicated calculation was given by Pumphrey and Bowkett⁽⁶⁾, and the sources of error were discussed⁽⁷⁾. The beam axis so determined was shown experimentally to be accurate to $\pm 0.05^\circ$ by comparing many solutions for beam direction from the same pattern. Another method in which matrix algebra was used⁽¹³⁾ was found accurate to $\pm 0.1^\circ$. The three-pole solution employing stereographic analysis^(1,2,9,10) and the graphical one-pole solution given by Heimendal⁽⁸⁾ were considered to be less accurate.

In this paper some alternative simple methods of beam axis determination, i.e., the three-normal solution and the combined pole-normal solutions, are described. The accuracy of these methods, including the three-pole solution, was evaluated by determining the angular divergence of ten or more beam axes solutions obtained from the same pattern.

BEAM AXIS SOLUTIONS FROM KIKUCHI PATTERNS

When three or more Kikuchi poles (K-poles) are recognizable or can be inferred from a diffraction pattern, the crystal orientation, i.e., the beam direction (anti-parallel to the transmitted electron beam) referred to the crystal coordinate axes, may be determined from a three-pole (3-P) solution. A schematic example is given in Figure 4.1a. Three K-poles are indexed and three simultaneous equations are formulated. The three pole equations for the 3-P solution are

$$\bar{P}_i \cdot \bar{B} = \cos \gamma_i, \quad i = 1, 2, 3 \quad (1a)$$

$$\text{or } h_i h + k_i k + l_i l = L / (L^2 + p_i^2)^{1/2}, \quad i = 1, 2, 3 \quad (1b)$$

where

$\bar{P}_i = [h_i k_i l_i]$ = a unit vector along the i^{th} pole axis,

$\bar{B} = [hk\ell]$ = a unit vector anti-parallel to the transmitted beam,

γ_i = angle between $\bar{P}_i$ and $\bar{B}$,

p_i = distance between the center of the transmitted spot and the i^{th} pole, and

L = the camera length (often called the effective camera length).

The angles γ_i and the camera length L are determined from an angle-distance calibration constant, which in turn is obtained from measurement of K-pole or Kikuchi pair (K-pair) separation made upon the diffraction pattern. When equations (1b) are employed, only positive values of the square roots are used in the calculation.

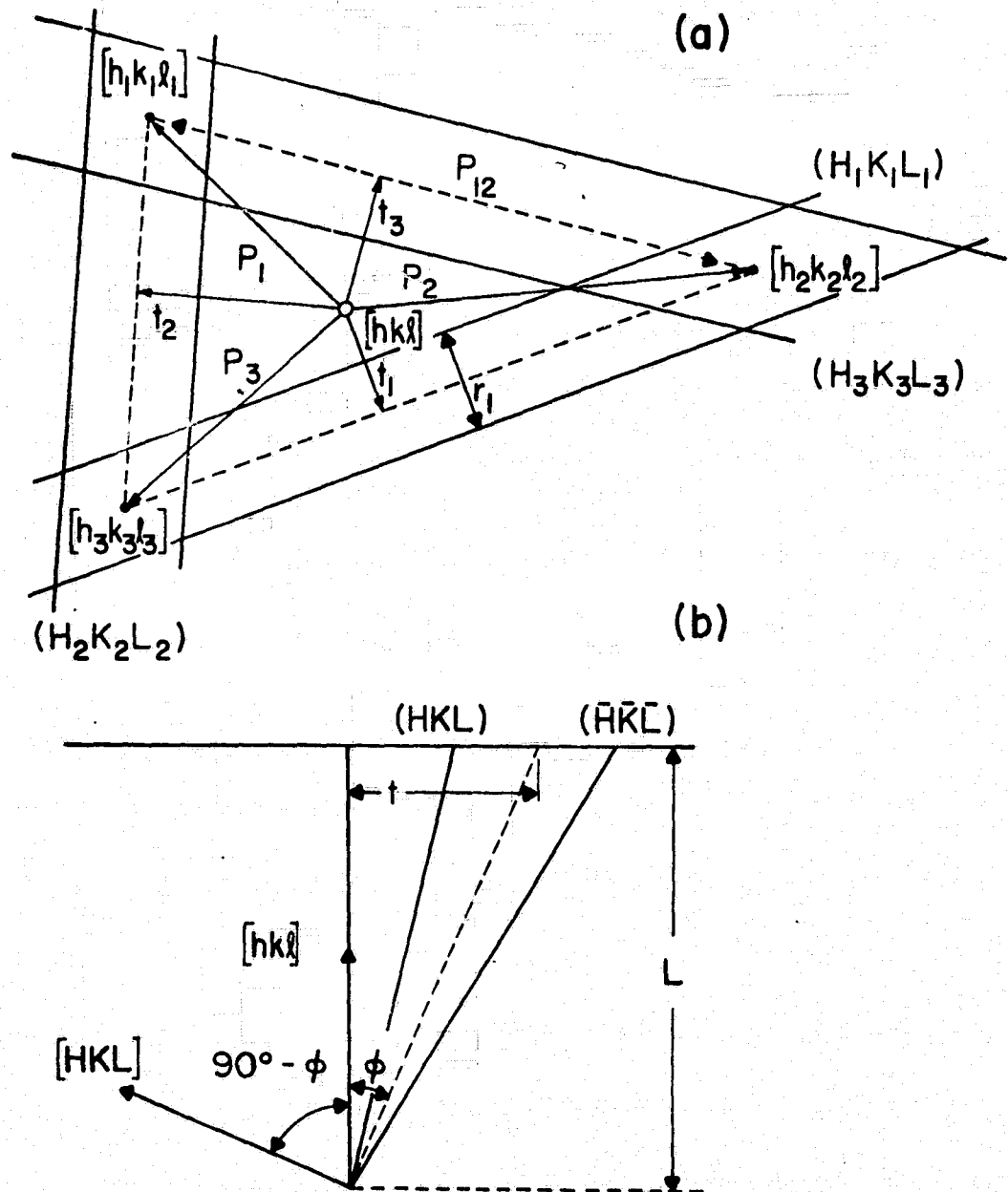


Fig. 4.1 (a) Schematic Kikuchi pattern showing the parameters to be measured for beam axis solution. (b) Diagram illustrating the formulation of normal equation using the trace distance t .

The beam axis can also be solved by a three-normal (3-N) solution, provided that three independent K-pairs which do not share the same K-pole are observed on the pattern. Kikuchi lines (K-lines) must first be indexed unambiguously and then three simultaneous equations can be formulated. As is seen from Figure 4.1a and 4.1b, the three normal equations for the 3-N solution are

$$\bar{N}_i \cdot \bar{B} = \cos(90^\circ - \phi_i) = \sin \phi_i, \quad i = 1, 2, 3 \quad (2a)$$

$$\text{or } H_i h + K_i k + L_i l = t_i / (L^2 + t_i^2)^{1/2}, \quad i = 1, 2, 3 \quad (2b)$$

where $\bar{B}$ and L are previously defined, and

$N_i = [H_i K_i L_i]$ = a unit vector along the normal of the $(H_i K_i L_i)$ plane,

ϕ_i = the angle between $\bar{N}_i$ and $\bar{B}$, and

t_i = the perpendicular distance between the transmitted spot and the trace of the $(H_i K_i L_i)$ net plane.

Again, ϕ_i and L are obtained from measurement of K-pole or K-pair separation, and only positive values of the square roots are used in equations (2b). It is to be noted that for each of the three normal equations, the indices of the K-line which is closer to the transmitted spot, i.e., the dark line of the pair on the printed pattern, is used to establish the equation. For the case where the net plane passes through the transmitted spot, indices of either K-line of the pair can be used. With this approach, the angles $(90^\circ - \phi_i)$ are always less than or equal to 90° so that negative dot products do not occur. It should also be pointed out that two possible beam axis solutions will be obtained from a set of three equations formulated from three K-pairs which share the same K-pole, since one of the three equations is a dependent equation. This arises

from the fact that the three normal vectors are co-planar, and the determinant of the coefficients of the three equations thus formulated is always equal to zero.

The angles ϕ_i are normally less than 10° , and the 3-N solution can be approximated such that

$$\bar{N}_i \cdot \bar{B} = \tan \phi_i \quad \text{small } \phi_i \quad (3a)$$

$$\text{or } H_i h + K_i k + L_i \ell = t_i / L \quad L \gg t_i \quad (3b)$$

The direction of beam axis $[hkl]$ obtained from this "approximate 3-normal" (A-3-N) solution is independent of L , and only the magnitude of the beam axis vector changes linearly with L . In fact, the equation (3b) can be rearranged into

$$H_i [hL] + K_i [kL] + L_i [\ell L] = t_i$$

The beam axis direction $[hL, kL, \ell L]$ thus determined is subject to the accuracy of the simplifying assumptions made. It will be shown later, however, that this A-3-N solution may be applied to all practical cases while preserving essentially the same accuracy as the 3-N solution.

It should be noted that a unique solution for the crystallographic direction of the beam axis requires simultaneous solution of three equations. Since the beam axis is related to the details of a diffraction pattern through the camera length, L , it would appear to be mathematically necessary for such simultaneous solutions to employ a precisely correct value of L . Use of an incorrect value of L would seem to produce no meaningful solution for the beam axis indices and would result in a magnitude of beam axis vector, $\bar{B}$, which is not equal to unity.

It must be realized, however, that any method of determining L is subject to uncertainty, at least from measurement errors, and an exact value of L cannot be found. Secondly, the simultaneous solution of the three required equations will produce a single beam axis solution, even when seriously deficient values of L are used. Consequently, the ability to obtain a final beam axis result does not provide the operator with any information concerning the accuracy of the solution. In the discussion which follows, the camera length will therefore be treated as a variable so that an assessment of the effect of uncertainties in L on the accuracy of various types of solutions can be made.

The beam axis may also be solved from three equations consisting of either (1) two pole equations and one normal equation (2-P/1-N) or (2) one pole equation and two normal equations (1-P/2-N). In these pole-normal solutions all combinations of poles and normals may be used. Unambiguous identification of K-lines is again required for unique beam axis determination. The one pole solution of Otte et al. ⁽³⁾ is essentially a 1-P/2-N solution, in which two K-pairs and the K-pole shared by the two K-pairs are used to formulate the 1-P/2-N equations. In their approach, K-pairs (not individual K-lines) are indexed, and negative values for the square roots in the two normal equations are allowed, thus leading to two possible solutions. By concluding that the third equation, the pole equation, was a dependent equation and gave no further information except for checking the accuracy of the solutions, Otte et al. employed a complicated procedure to calculate the two possible solutions from the two normal equations. A second K-pole is therefore required if the determination is to be unique. Careful analysis reveals that the three equations in the 1-P/2-N solution are actually independent, since the two normals and the pole axis are not co-planar. If individual K-lines which belong to the K-pole are indexed unambiguously

and appropriate K-lines are used to formulate the equations, a unique solution can be easily obtained. With the help of Kikuchi projections (17-19) or Kikuchi maps (2, 20), unambiguous indexing of a pattern with only one K-pole can usually be carried out without difficulty. In fact, this presents one of the important uses of Kikuchi projections or maps.

The behavior of the five beam axis solutions mentioned above, i.e., the 3-P, the 3-N, the A-3-N, the 2-P/1-N, and the 1-P/2-N solutions, was examined in this study using the type of equations given in (1b), (2b), and (3b). The error involved in such solutions arises mainly from pattern distortion and errors in p_i , t_i , and L . Pattern distortion comes from lens aberration, lattice strain, and any distortion from photographic processing. The error which arises from pattern distortion and from measurement of p_i and t_i can not be systematically eliminated, and is therefore considered to be the inherent error. The error which results from uncertainty in L , or in γ_i and ϕ_i if equations (1a), (2a), and (3a) are used, can be significantly large. These two errors will be evaluated in this study. One method of evaluation is to determine the angular spread of many beam axes obtained from the same pattern for a range of values of L . The angular spread is then taken as a measure of accuracy of each method. In this study, measurement of distances made upon K-patterns was performed using an ordinary metric ruler. The calculations were carried out using an IBM 360 computer. It should be noted that equal accuracy will be assumed for equations (1a), (2a), and (3a) based on the premise that γ_i , ϕ_i , and L are all determined in the same manner, i.e., from distance of separation of known K-poles or K-pairs.

The exact equation for determination of L from K-pole separation is

$$\cos \gamma_{ij} = (2L^2 + p_i^2 + p_j^2 - p_{ij}^2) / 2[(L^2 + p_i^2)(L^2 + p_j^2)]^{1/2} \quad (4a)$$

where γ_{ij} and p_{ij} are the angle and distance between $[h_i k_i \ell_i]$ and $[h_j k_j \ell_j]$ poles, respectively. The exact equation for computing L from K -pair separation is

$$\cos 2\theta_i = (L^2 + t_i^2 - \frac{1}{4}r_i^2) / \{ [L^2 + (t_i + \frac{1}{2}r_i)^2] [L^2 + (t_i^2 - \frac{1}{2}r_i)^2] \}^{\frac{1}{2}} \quad (4b)$$

where θ_i and r_i are the Bragg angle and K -pair separation of $(H_i K_i L_i)$ plane, respectively. The angle θ_i is determined from the Bragg equation. The solution for L using equation (4a) or (4b) is tedious, and it can only be solved graphically. The approximate equations generally used for solving L are

$$\tan \gamma_{ij} = p_{ij}/L \quad (5a)$$

and

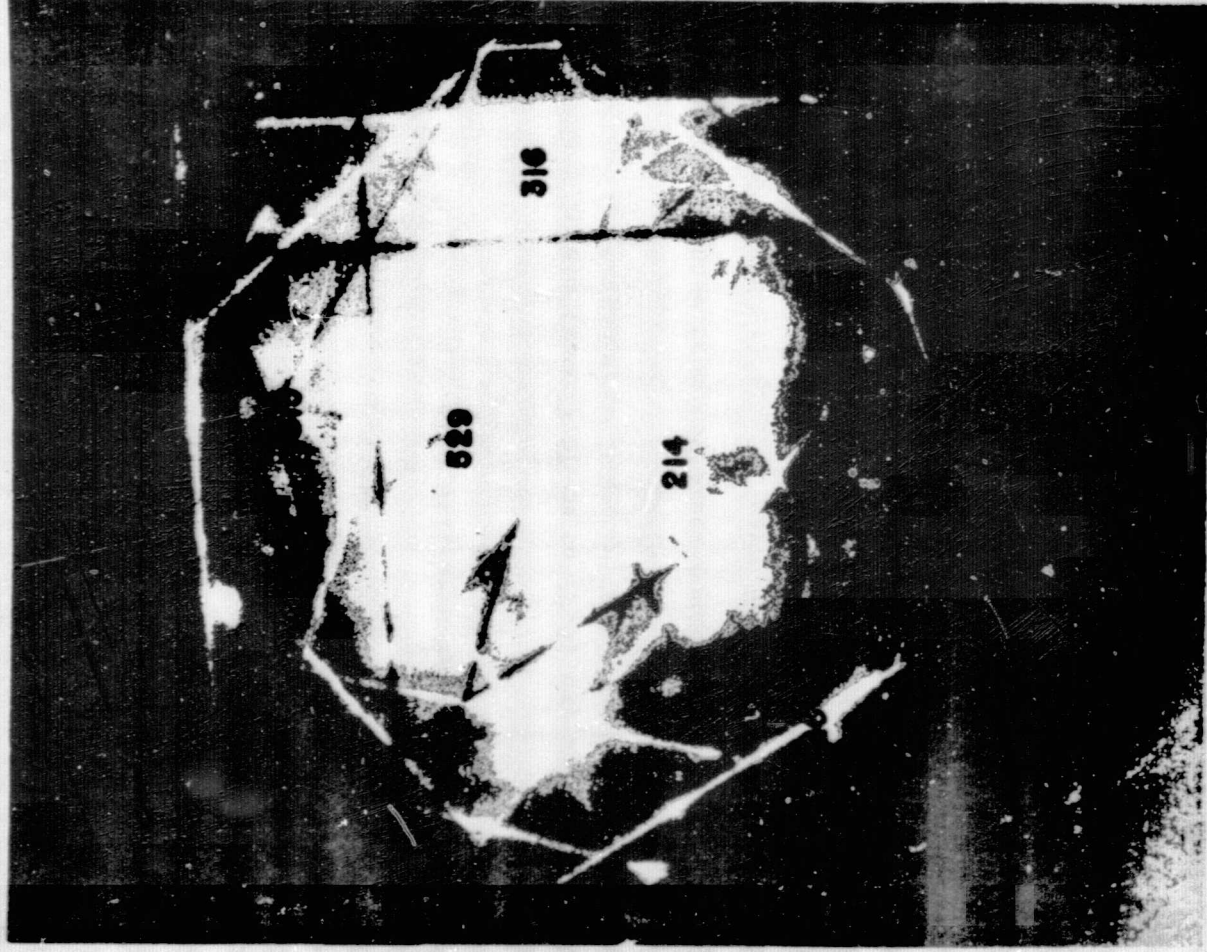
$$r_i d_i = \lambda L \quad (5b)$$

where d_i is the d -spacing of $(H_i K_i L_i)$ plane, and λ is the electron wavelength.

RESULTS AND DISCUSSION

Figure 4.2 is one of three K-patterns used to evaluate the accuracy of beam axis solutions. The pattern is seen to be slightly distorted at the outer edges. To evaluate the accuracy of the five beam axis solutions, the value of L was first determined from an 8" x 10" enlarged print of the pattern shown in Figure 4.2. Three values of L were calculated using the exact equation (4a). They ranged from 77.37 cm to 79.05 cm, with an average value of 78.41 cm. This average value will be used as the most accurate value of L . Another ten values of L were determined using the approximate equation (5a). They spread from 77.57 cm to 79.31 cm, with an average value of 78.48 cm. It is seen that the values of L obtained from equation (5a) are in good agreement with those from the exact equation (4a), and are accurate to $\pm 1\%$. Also, another ten values of L were calculated using equation (5b). They varied from 72.83 cm to 75.90 cm, with an average of 74.85 cm. It is noted that the values of L thus determined are 5-7% less than the true L . This large discrepancy is believed to be due to uncertainties in electron wave length and lattice parameters, and uniform lattice strain existing within the thin foil crystal prepared for electron microscopy. The results presented herein were obtained from the pattern shown in Figure 4.2. The two other patterns examined were used to verify if the results obtained were equally applicable to other diffraction patterns. Those results fell within the accuracy reported here.

Ten 3-P solutions, twelve 3-N solutions, twelve A-3-N solutions, twelve 2-P/1-N solutions, and twelve 1-P/2-N solutions were generated at $L = 78.41$ cm for the beam axis shown in Figure 4.2. The most accurate beam axis solution for each of the five techniques was taken from the average of the solutions



$(\bar{5}\bar{1}3)$

$(40\bar{2})$

$(\bar{4}22)$

$(\bar{3}31)$

$(\bar{2}60)$

$(\bar{2}40)$

$(13\bar{1})$
 $(1\bar{2}1)$

(062)

(353)
 (242)

(533)

obtained for that type. This involves the assumption that the inherent errors tend to scatter such calculations in all directions away from the true beam axis, so that the most probable value is at the center of what approximates a solid cone of calculated beam axes distributed about the mean value. The K-poles and K-lines used for the ten or more solutions from each of the five techniques were deliberately taken from the full range of possibilities around the center of the diffraction pattern, so as to maximize the validity of this assumption. The five average solutions obtained using $L = 78.41$ cm were found to deviate by less than 0.02° from the average of the five. This represents a distance approximately equal to the resolution of the human eye on the enlarged photographic print. For this reason, the average beam axis for each type of solution was used as the most accurate solution of that group.

With the most accurate solutions thus determined, the beam axis calculations were repeated using the assumed L values ranging from 70 to 86 cm. For each value of L used, the angular deviations of the calculated beam axes as compared with the most accurate solution of the same method were computed. The maximum deviations were interpreted as the probable errors associated with the particular method employed. The magnitude of the beam axis vector, $\sqrt{S}$, where $S = h^2 + k^2 + \ell^2$, was also computed for each solution. For the 3-N and the A-3-N solutions, calculations were further carried out with values of L ranging from 1 to 1000 cm, since these techniques were found to be highly insensitive to such variations. The results obtained are listed in Tables I-IV. The maximum angular deviations are plotted vs. the assumed value of L in Figure 4.3. It is seen that the 3-N and the A-3-N methods are the most consistent and hence the most accurate.

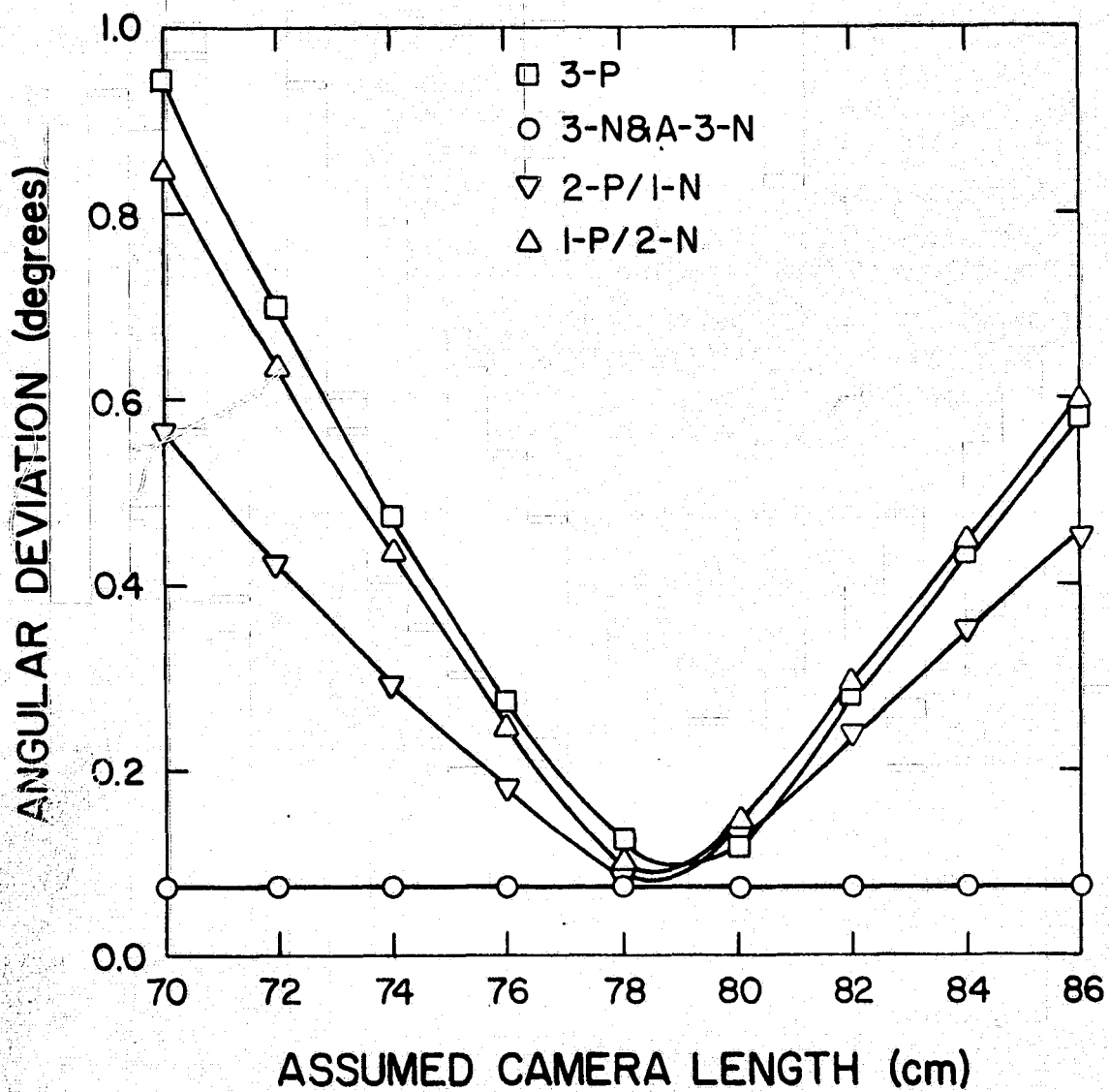


Fig. 4.3 Angular divergence of various beam axis solutions as compared with the most accurate solutions calculated using the true camera length.

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obtained for that type. This involves the assumption that the inherent errors tend to scatter such calculations in all directions away from the true beam axis, so that the most probable value is at the center of what approximates a solid cone of calculated beam axes distributed about the mean value. The K-poles and K-lines used for the ten or more solutions from each of the five techniques were deliberately taken from the full range of possibilities around the center of the diffraction pattern, so as to maximize the validity of this assumption. The five average solutions obtained using $L = 78.41$ cm were found to deviate by less than 0.02° from the average of the five. This represents a distance approximately equal to the resolution of the human eye on the enlarged photographic print. For this reason, the average beam axis for each type of solution was used as the most accurate solution of that group.

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TABLE I

Angular Divergence of Beam Axes From the 3-P Solution (Degrees)
For Various Assumed Values of L (cm)

K-Poles Used for 3-P Solutions	L = <u>70</u>	<u>74</u>	<u>78.41</u>	<u>82</u>	<u>86</u>
[316] [529] [214]	0.508	0.287	0.0815	0.062	0.201
[316] [326] [529]	0.803	0.436	0.0970	0.148	0.378
[315] [316] [326]	0.130	0.087	0.0814	0.103	0.135
[213] [417] [214]	0.946	0.473	0.0431	0.280	0.579
[213] [417] [327]	0.303	0.148	0.0339	0.110	0.206
[315] [215] [326]	0.230	0.105	0.0582	0.126	0.205
[213] [315] [214]	0.791	0.387	0.0187	0.253	0.509
[417] [327] [326]	0.096	0.065	0.0811	0.111	0.145
[215] [326] [529]	0.573	0.273	0.0557	0.219	0.407
[215] [327] [529]	0.507	0.227	0.0383	0.219	0.396
Average Divergence	0.489	0.249	0.059	0.163	0.316
Magnitude of Beam Axis Vector, $\sqrt{S}$	0.9995	0.9997	1.0000	1.0002	1.0004

TABLE II

Angular Divergence of Beam Axes From the 3-N and the A-3-N
Solutions (Degrees) for Various Assumed Values of L (cm)

K-Lines used for 3-N and A-3-N Solutions	L = $\frac{20}{(3-N)}$	$\frac{40}{(3-N)}$	$\frac{70-86}{(3-N)}$	$\frac{1000}{(3-N)}$	$\frac{A11 L}{(A-3-N)}$
[$\bar{3}31$] [$40\bar{2}$] [$24\bar{2}$]	0.007	0.010	0.010	0.010	0.010
[$13\bar{1}$] [$40\bar{2}$] [$2\bar{4}0$]	0.012	0.012	0.018	0.020	0.020
[$0\bar{6}2$] [$40\bar{2}$] [$\bar{3}31$]	0.052	0.011	0.023	0.028	0.027
[$24\bar{2}$] [$\bar{2}60$] [$40\bar{2}$]	0.004	0.011	0.014	0.015	0.015
[$24\bar{2}$] [$1\bar{5}1$] [$\bar{4}22$]	0.086	0.064	0.058	0.056	0.057
[$53\bar{3}$] [$1\bar{5}1$] [$\bar{3}31$]	0.027	0.026	0.027	0.027	0.026
[$\bar{4}22$] [$13\bar{1}$] [$2\bar{4}0$]	0.084	0.074	0.073	0.072	0.073
[$\bar{5}1\bar{3}$] [$13\bar{1}$] [$2\bar{4}0$]	0.026	0.009	0.011	0.013	0.013
[$31\bar{1}$] [$53\bar{3}$] [$13\bar{1}$]	0.150	0.080	0.074	0.074	0.074
[$\bar{5}1\bar{3}$] [$13\bar{1}$] [$\bar{3}31$]	0.039	0.005	0.005	0.008	0.008
[$1\bar{5}1$] [$\bar{4}22$] [$53\bar{3}$]	0.081	0.059	0.053	0.052	0.052
[$40\bar{2}$] [$2\bar{4}0$] [$\bar{3}5\bar{3}$]	0.009	0.012	0.013	0.014	0.013
Average Divergence	0.048	0.031	0.032	0.033	0.032
Magnitude of Beam Axis Vector, $\sqrt{S}$	3.8577	1.9656	1.1282- 0.9189	0.0791	1.0093

TABLE III

Angular Divergence of Beam Axes from 2-P/1-N Solution
(Degrees) for Various Assumed Values of L (cm)

K-Poles and K-Lines used for 2-P/1-N Solutions	L = <u>70</u>	<u>74</u>	<u>78.41</u>	<u>82</u>	<u>86</u>
[529] [214]/[24 $\bar{2}$]	0.113	0.068	0.030	0.021	0.041
[529] [316]/[3 $\bar{3}$ 1]	0.492	0.261	0.044	0.111	0.262
[417] [213]/[4 $\bar{2}$ 2]	0.507	0.238	0.042	0.220	0.412
[326] [316]/[40 $\bar{2}$]	0.179	0.092	0.030	0.067	0.125
[327] [529]/[24 $\bar{2}$]	0.385	0.175	0.027	0.159	0.292
[213] [327]/[1 $\bar{5}$ 1]	0.525	0.289	0.062	0.104	0.269
[215] [417]/[13 $\bar{1}$]	0.487	0.263	0.075	0.152	0.310
[316] [529]/[24 $\bar{0}$]	0.516	0.267	0.035	0.131	0.292
[315] [214]/[13 $\bar{1}$]	0.564	0.292	0.040	0.165	0.354
[316] [326]/[4 $\bar{2}$ 2]	0.561	0.265	0.039	0.240	0.453
[417] [327]/[40 $\bar{2}$]	0.215	0.109	0.026	0.079	0.152
[316] [315]/[1 $\bar{5}$ 1]	0.425	0.240	0.073	0.102	0.231
Average Divergence	0.414	0.213	0.043	0.129	0.266
Magnitude of Beam Axis Vector, $\sqrt{S}$	0.9994	0.9997	1.0000	1.0002	1.0004

TABLE IV

Angular Divergence of Beam Axes From 1-P/2-N Solution
(Degrees) for Various Assumed Values of L (cm)

K-Poles and K-Lines used for 1-P/2-N Solutions	L = <u>70</u>	<u>74</u>	<u>78.41</u>	<u>82</u>	<u>86</u>
[214]/[40 $\bar{2}$] [2 $\bar{4}$ 0]	0.136	0.067	0.002	0.049	0.100
[529]/[$\bar{3}$ 31] [24 $\bar{2}$]	0.238	0.128	0.020	0.060	0.141
[316]/[13 $\bar{1}$] [$\bar{3}$ 31]	0.505	0.287	0.055	0.110	0.277
[316]/[13 $\bar{1}$] [40 $\bar{2}$]	0.509	0.282	0.060	0.108	0.274
[417]/[13 $\bar{1}$] [4 $\bar{2}$ 2]	0.653	0.330	0.077	0.251	0.486
[417]/[$\bar{3}$ 51] [5 $\bar{1}$ 3]	0.655	0.329	0.013	0.229	0.469
[215]/[42 $\bar{2}$] [2 $\bar{4}$ 0]	0.663	0.313	0.032	0.286	0.544
[327]/[3 $\bar{1}$ $\bar{1}$] [1 $\bar{5}$ 1]	0.582	0.284	0.069	0.247	0.465
[213]/[4 $\bar{2}$ 2] [0 $\bar{6}$ 2]	0.847	0.434	0.080	0.295	0.596
[214]/[1 $\bar{5}$ 1] [40 $\bar{2}$]	0.417	0.226	0.039	0.101	0.241
[316]/[$\bar{3}$ 31] [24 $\bar{2}$]	0.239	0.128	0.020	0.060	0.141
[315]/[2 $\bar{4}$ 0] [40 $\bar{2}$]	0.137	0.068	0.002	0.050	0.100
Average Divergence	0.465	0.239	0.039	0.154	0.319
Magnitude of Beam Axis Vector, $\sqrt{S}$	0.9999	0.9999	1.0000	1.0000	1.0000

The beam axes obtained from the 3-N solution are nearly independent of assumed L greater than 40 cm, and the accuracy is affected only when L is assumed to be less than 40 cm. The beam directions determined from the A-3-N method are as accurate as those from the 3-N solution, and are essentially invariant to the change of L . In each of these two methods, the maximum angular deviations obtained were 0.07° , and the average for each was only 0.032° . These average deviations are insensitive to variations of L , and are assumed to be the inherent errors for these two solutions. For the 3-P, the 2-P/1-N, and the 1-P/2-N methods, the angular deviations all become minimum near $L = 78.41$ cm, and increase as the assumed error in L increases. The inherent errors, taken to be the average divergence at $L = 78.41$ cm, are 0.059° , 0.043° , and 0.039° for the 3-P, 2-P/1-N, and 1-P/2-N methods, respectively. Based on the fact that L can be determined from the present example to within 1% from K-pole separation and to within 5-7% from K-pair separation, it is seen that for those solutions with at least one pole equation applied, the beam axes can be obtained to 0.14° if distance between K-poles is used to calculate L , and to 0.7° if distance of K-pair separation is used to compute L . Results of the variation in magnitude of beam axis vector, $\sqrt{S}$, indicate that beam axes from the pole and pole-normal methods will not deviate largely from unity for large variations in L , whereas the direction will experience significant variation. With the normal methods, however, large variations in L produce little or no change in the direction of the computed beam axes while the magnitude of the beam axis vector varies significantly with changes in L . Therefore, the A-3-N and the 3-N solutions are suggested whenever three independent K-pairs are recognizable or can be inferred from the pattern.

In general, it has been found that the angles ϕ_1 encountered in transmission electron microscopy are less than 10° , e.g., in this study they were less than 7° . The approximation used in A-3-N solution has been found to be of sufficient accuracy for ϕ_1 less than 10° so that no significant loss of accuracy occurs in using this method in practical cases. Consequently, it may be concluded that the A-3-N solution of equation (3c) is the simplest method which can be used with high accuracy.

The camera length is generally not a constant. As the specimen position changes along the electron-optical axis during specimen tilting, a refocusing of the image using the objective lens or a repositioning of specimen using the "z" adjustment, if available, is required. The refocusing using the objective lens can cause a significant variation in camera length, as well as in magnification, at a constant intermediate lens setting for the same projection pole piece used. There is normally a change of 30%/mm of specimen shift for modern short focus objective lenses⁽¹⁶⁾. For the Siemens Elmiskop 1A electron microscope used in this study, an increase of 35% in camera length and a decrease of 30% in magnification were observed as the specimen was raised by a distance of 1 mm. With the Swann biaxial tilting stage which provides for a maximum tilt of $\pm 20^\circ$, a variation as much as 20% in both camera length and magnification is possible. Therefore, the camera length must be determined for each pattern if the 3-P, the 2-P/1-N, or the 1-P/2-N solution is used. On the other hand, any approximate value of L, not less than half of the true L, may be used if the 3-N solution is employed.

While it might be argued that placing the beam axis precisely at a known Kikuchi pole would provide a better reference for the most accurate solution, it should be noted that at least two shortcomings would result. First,

the inherent accuracy of the various solutions averages about 0.04° , and this represents less than 0.2 mm on the fluorescent screen during normal operation; i.e., it would be quite difficult to set the beam precisely at the center of a Kikuchi pole with the kind of accuracy needed to exceed that of the solutions presented here. Secondly, when a Kikuchi pole is on the beam axis, the dynamical effects in broadening the Kikuchi lines are such as to make definition of the pole location inherently uncertain. This problem is significantly diminished when Kikuchi poles used are away from the beam axis as was done in the present analysis.

Appendix C

Computer Generation and Identification
of Divergent Beam Diffraction Phenomena

by
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COMPUTER GENERATION AND IDENTIFICATION
OF DIVERGENT BEAM DIFFRACTION PHENOMENA

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Abstract

A computer program has been developed to provide for computer plotting of standard stereographic projections of divergent beam diffraction phenomena; i.e., Kossel, pseudo-Kossel, Coates, and Kikuchi patterns. A previous version of this program could provide plots only for cubic and hexagonal crystals and used tabular angle-distance values to identify diffraction lines. The revised program uses lattice parameters, lattice angles, and atomic positions to generate the appropriate diffraction information for any desired crystal lattice. The new program eliminates the more tedious angle-distance tabular identification of diffraction lines by identifying the lines directly around the edge of the line projection. Provision has also been made for optional plots of plane normal stereographic projections, zone axis stereographic projections, Wulff nets for use with the stereographic projections, and spot diffraction patterns for transmitted electron microscopy.

The program has now been adapted to a user-interactive computer graphics terminal. The basic aspects of the program will be described and typical examples of diffraction simulations will be presented.

Introduction

The line diffraction patterns which result when a divergent beam diffracts in a crystal are observed as either: (1) the Kikuchi lines found in transmission electron microscopy (1-3), (2) the Coates patterns generated in scanning electron microscopy (4-6), or (3) the diffraction conics observed in Kossel or pseudo-Kossel X-ray studies (7-12). In each case, the lines are used for various analytical purposes, e.g., the accurate determination of crystal orientation with respect to the incident beam. The major drawback of these analytical approaches is that they are tedious, especially in non-cubic crystals, and are subject to error. This situation may be corrected through the use of computer graphics. The following paper describes a computer program which was written in FORTRAN language for the CALCOMP plotter.

Results and Discussion

The original version of this program consisted of two card decks; one for three cubic crystals (face-centered cubic, body-centered cubic, and diamond cubic), and one for hexagonal close-packed crystals. That version of the program has been published elsewhere and should be referenced for the details of the matrix algebra and stereographic projection geometry, since they will not be repeated here (13).

The program described herein began with the former program as a base but has been extensively revised to provide more general utility and eliminate the somewhat tedious angle-distance listings which were formerly required for identification of diffraction conics. The new program consists of a single deck of 1592 cards, requires about 160K of storage space, and will generate the diffraction lines which are allowable by the structure factor for any desired unit cell based on the following input parameters;

1. The unit cell dimensions (A, B, and C)
2. The three lattice angles (AA, BA, CA)
3. The positions of the atoms (only one type of atom is included)
4. The crystallographic direction desired for the incident beam taken pointing toward the viewer (the X3 axis). The X3 axis can also be given as a plane normal.
5. The crystallographic direction which is intended to point toward the bottom of the plot (the X1 axis)
6. The wavelength in Angstroms of the radiation used (WL)
7. The radius of the stereographic projection sphere desired (PSR)
8. The set of {hkl} planes which the computer uses, in terms of interplanar spacings, to determine how many diffraction lines to plot (LHKL).

9. The type of plot desired, i.e., line projection (LLP), identification projection (LIP), plane normal projection (LNP), zone axis projection (LZP), Wulff net (LWN), or spot pattern (LSP). These plots will be described shortly and more than one may be requested in a given input deck.
10. The dimensions of the area which will contain the plot (XL and YL) and the length of paper to be assigned to each plot (PAL). The outer circle of the stereographic projection appears, either wholly or in part, when the plotting area is sufficiently large relative to PSR.
11. The location of the projection point for the stereographic projection, i.e., the location of the X3 axis within the plot frame. This is given in terms of the x and y coordinates from the center of the plot (OX and OY).
12. The name of the material or other identifying feature to be inserted in the fourth line of the figure title. The title block automatically supplied with each plot identifies, except in the case of Wulff nets, the type of plot and the values of X3, X1, WL, PSR, LHKL, AA, BA, CA, A, B, and C. It also states the value of MDZP generated within the program. For Wulff nets, only the type of plot and the number of degrees per division are given. The value of MDZP is the minimum number of intersecting pairs of corresponding diffraction lines in a line plot which will be used to trigger the identification of the corresponding zone axis in the identification projection (see also reference 13).

An example of a Kikuchi line projection for aluminum using a PSR of 51 cm is shown in Figure 1. The value of X3 was [025] and X1 was taken as $[\bar{2}0 \cdot 5 \cdot \bar{2}]$. In order to simulate the pattern for 100 KV electrons, a WL of 0.0370 Angstroms was used, and the identification projection (LIP) was called to be superimposed on the line projection (see also reference 13). In this case, LHKL = (551) was used to limit the number of lines drawn. In terms of the working details of the program, only allowable diffraction lines for planes whose interplanar spacings were equal to or greater than the (551) were plotted. If LHKL = (440) were used, Figure 1 would contain about 50% fewer lines.

The major new feature of Figure 1 relative to the former program is that the Kikuchi lines are identified around the edges of the plot. The program prevents redundancy in this labeling as well as overcrowding. A second row of labels is provided when needed if the plotting area is of sufficient size. Also note that the origin is marked by a square data symbol and is located at the intersection of the X1 arrow and the arrow at the right hand edge. The latter is the X2 vector (right-handed system) which need not be an input to the program but is used to assist the viewer in locating the origin.

To demonstrate that the program represents a powerful tool for simulation, a selected-area electron diffraction pattern obtained at 100 KV in aluminum is shown in

Figure 2 for which Figure 1 is the computer simulation. It should be noted that the experimental patterns are not stereographic projections but that for the larger values of PSR, the stereographic approximation is excellent.

The program is equally capable of simulating Kossel patterns when the appropriate wavelength is used. Figure 3 shows a Kossel pattern simulation for an iron crystal using $WL = 1.7902$ Angstroms (FeK_{α} radiation). For this pattern, $X3$ was taken as $[229]$, $X1 = [1\bar{1}0]$, $LHKL = (332)$, $PSR = 17$ cm, and the zone axis identification was again superimposed on the line projection.

Figure 4 shows a cubic stereographic projection of plane normals for $X3 = [211]$, $X1 = [\bar{1}11]$, and such plots are, of course, useful in many crystallographic studies; in transmission electron microscopy they are especially helpful in trace analysis.

The program has been recently modified to produce transmission electron diffraction spot patterns. This is done by simple geometric construction from the spacings and orientation of allowable Kikuchi line pairs which would be predicted for a given $X3$ at $PSR = 90$ cm. Figure 5 shows the simulated $[105]$ spot pattern for aluminum at 100 KV with $X1 = [010]$.

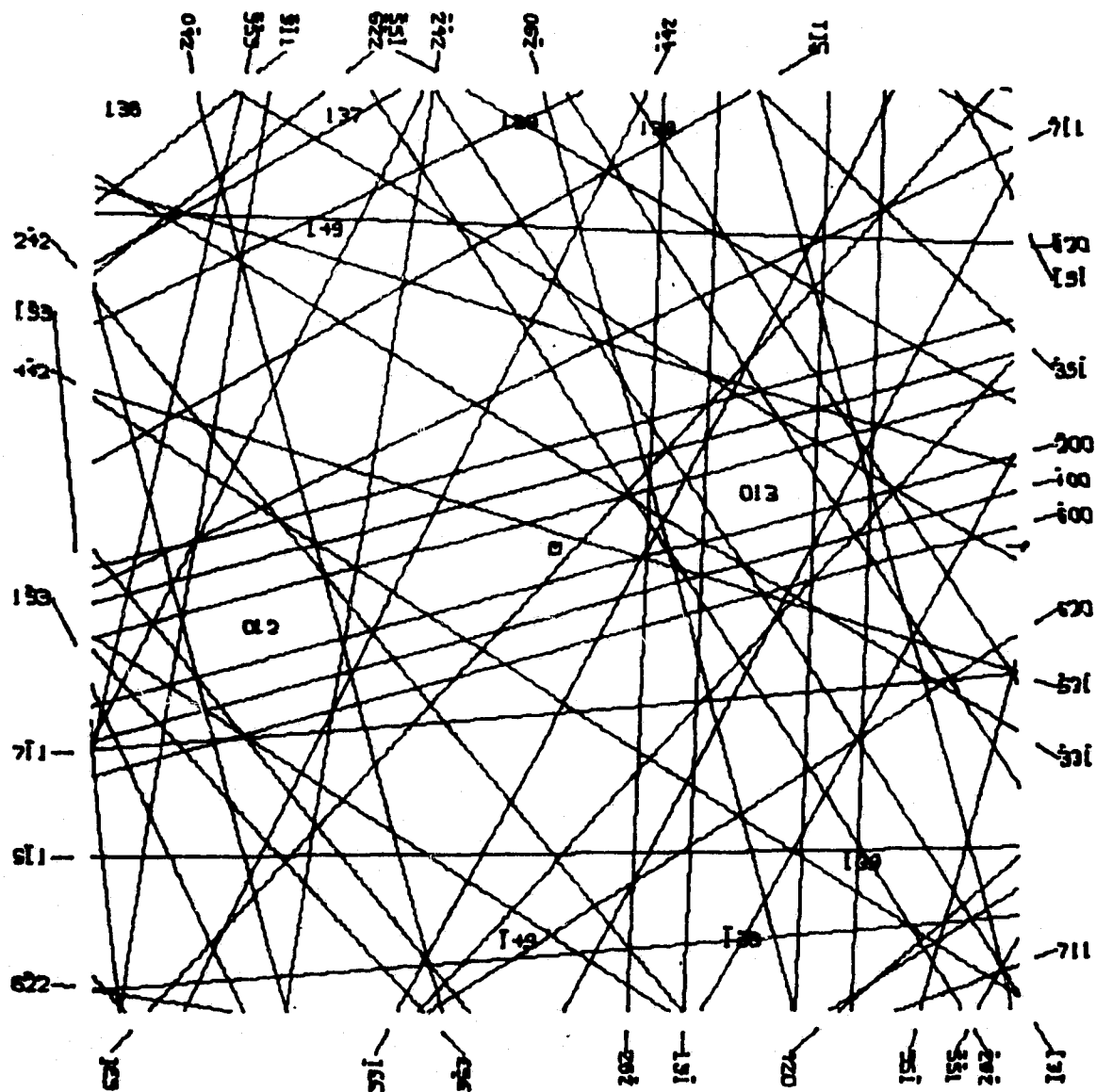
For enhanced speed of output and improved classroom utility, the program has been written onto a user-interactive computer graphics terminal in our laboratories. Using this approach, a student can obtain any desired plot in a few minutes and obtain a copy for analytical purposes from the hard copy unit connected to the terminal. The cost of such plots may be three dollars or less, depending on the complexity of the plot and the way priority charges are handled.

Acknowledgment

The authors are grateful to Mr. Sam Snyder whose diligent work adapted the FORTRAN program to the computer graphics terminal and added the capability for simulating spot patterns. The financial support of both the National Science Foundation and the NASA-Langley Research Center during various phases of program development is gratefully acknowledged.

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$\downarrow$
 X_1
 1 0 2 S 12 LINE PROJECTION
 WL. .0370 Å. XL. 1 .20 S .2 1 MOZP. 2
 PSR. 51.00 CM. LHKL. 1 S S 1 1
 ALUMINUM
 A. 4 0495 .B. 4 0495 .C. 4 0495 .AA. 90 00 .BA. 90 00 .CA. 90 00

FIG. 1

Standard stereographic line projection with superimposed identification projection for aluminum at 100 kV using PSR = 51 cm, $X_3 = [025]$, $X_1 = [20.5 \cdot 2]$, WL = 0.0370 Å, and LHKL = (551). The caption above was also drawn by the computer.

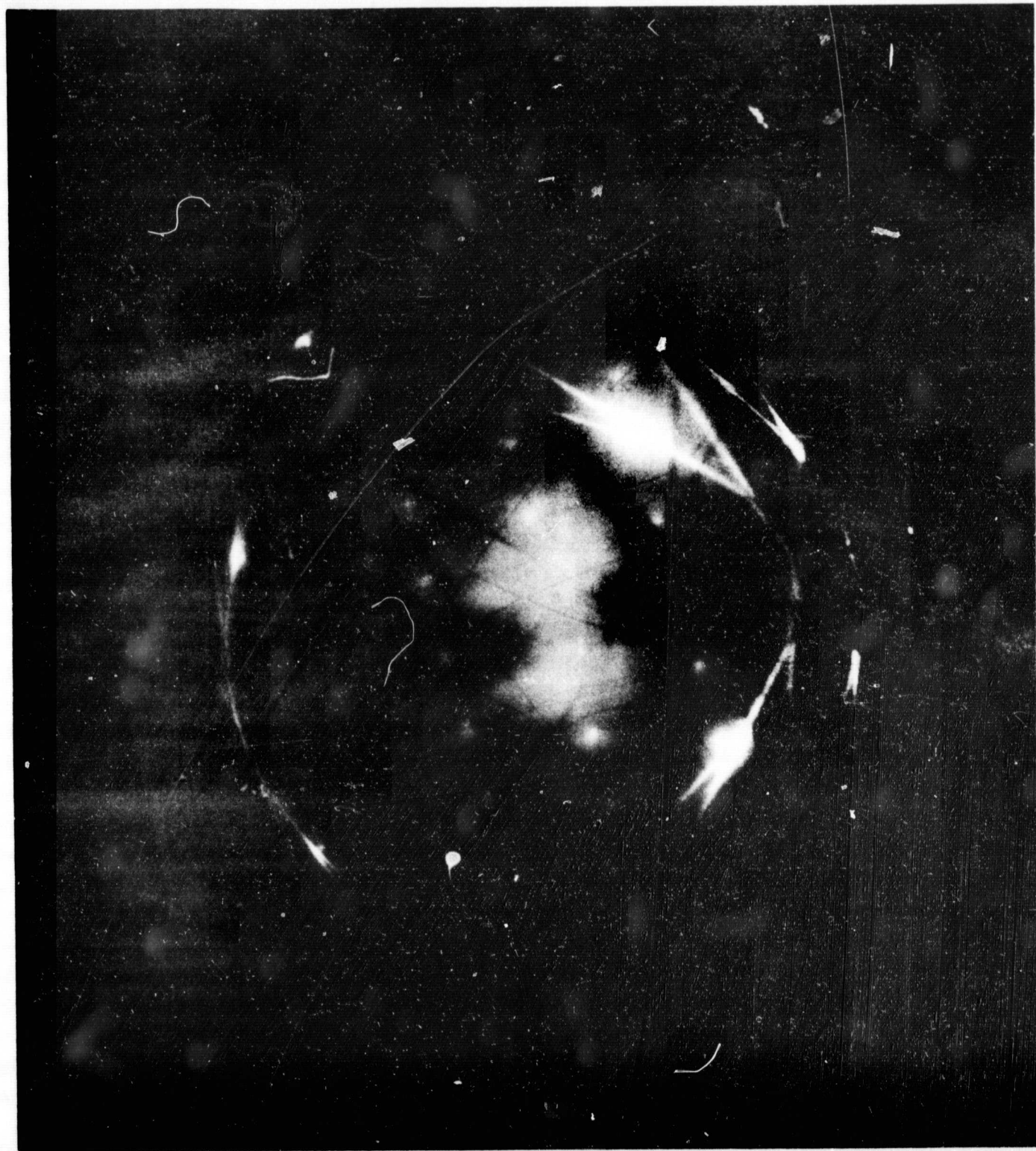


FIG. 2

Transmission electron diffraction pattern taken from aluminum foil at 100 kV. Beam orientation near [025].

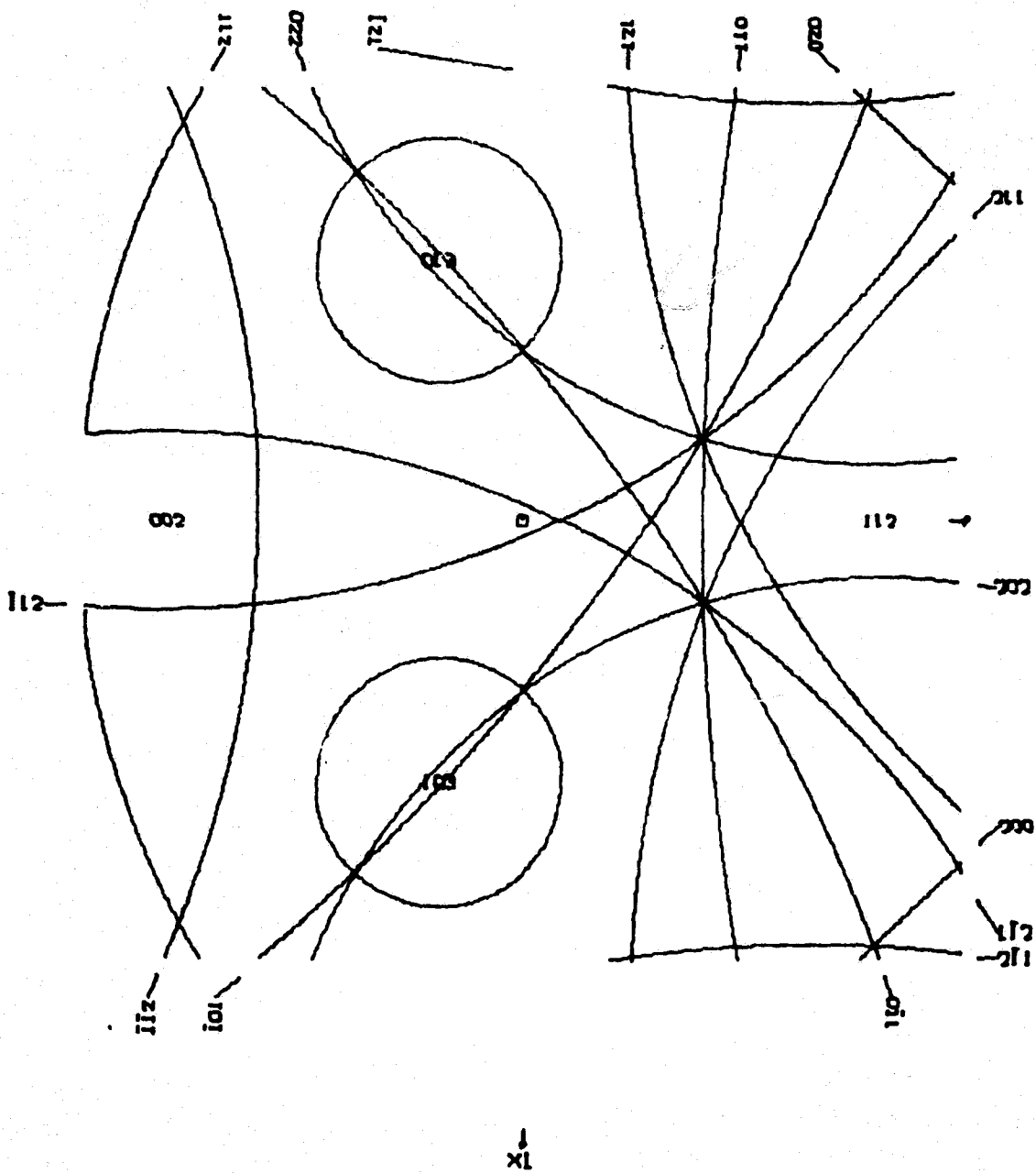


FIG. 3

Simulation of Kossel pattern for iron using FeK_α radiation. PSR = 17 cm, $X3 = [229]$, $X1 = [1\bar{1}0]$, and LHKL = $[332]$. Identification projection is included.

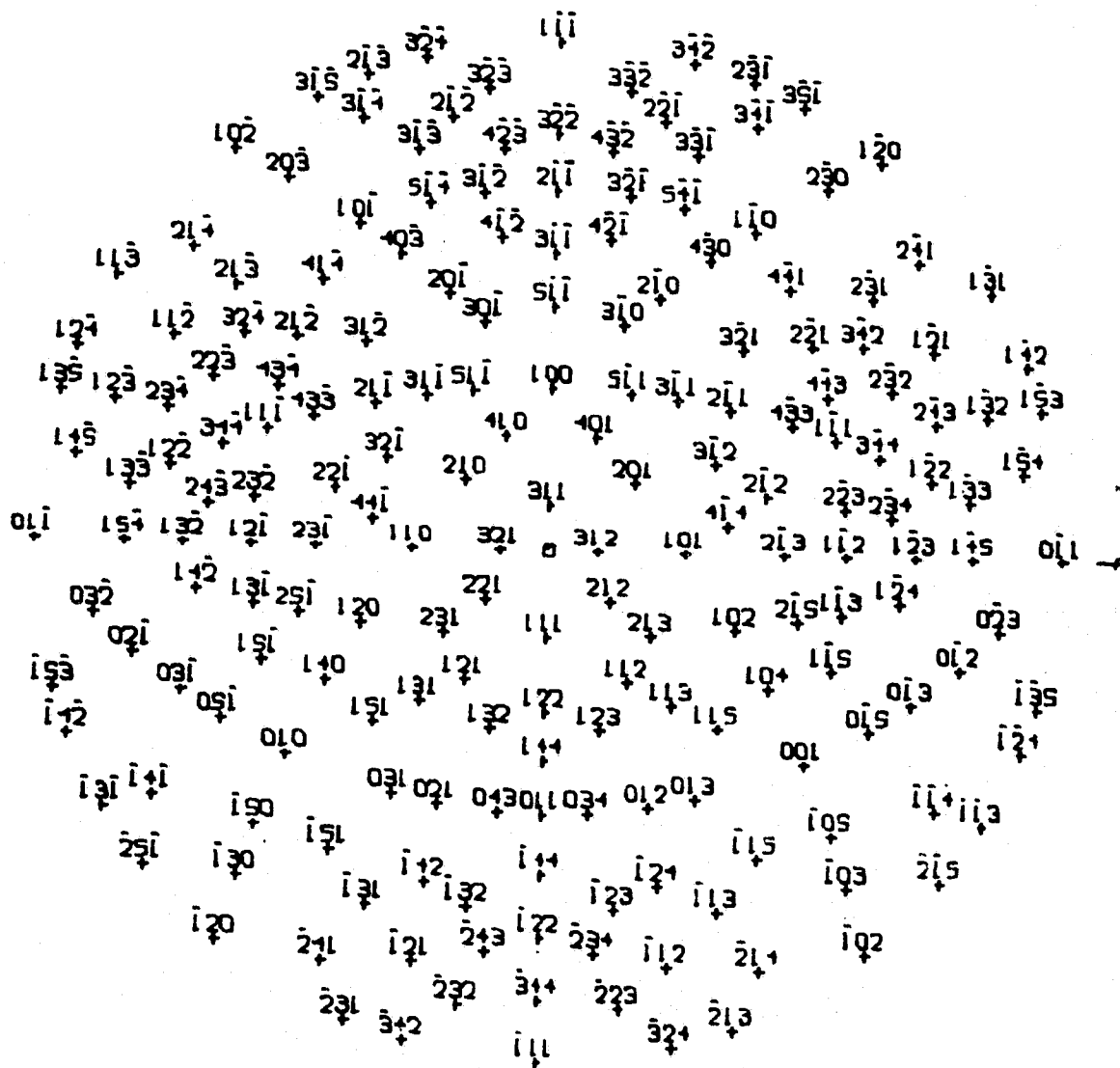
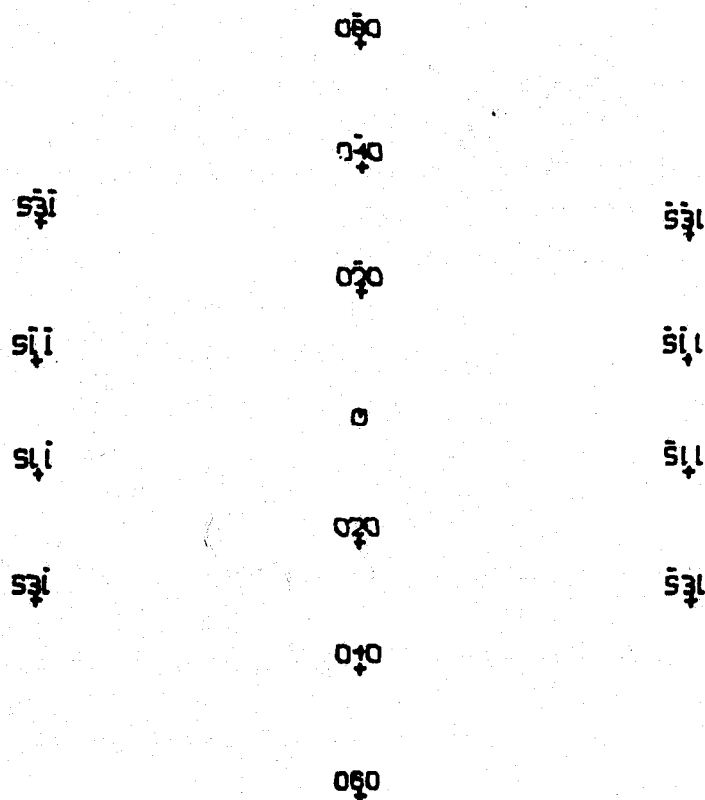


FIG. 4

Standard (211) cubic stereographic projection of plane normals for $X_3 = [211]$, $X_1 = [\bar{1}11]$, and $PSR = 3.5$ cm.



1 1 0 5 12 SPOT PROJECTION
 WL. .0370 A. XL 1 0 1 0 1 MOZP. 2
 PSR. 90.00 CM. LMKL. 1 8 0 0 1
 ALUMINUM
 A. 1.0495 .B. 1.0495 .C. 1.0495 .AA. 90.00 .BA. 90.00 .CA. 90.00

FIG. 5

Simulated [105] diffraction spot pattern for aluminum at 100 kV. PSR = 90 cm, X3 = [105], X1 = [010].

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CONCLUSIONS

It is concluded from the present results that among the five beam axis solutions presented the 3-N and the A-3-N methods are the most consistent and hence the most accurate. Beam axes so determined are accurate to 0.07° . The A-3-N solution, in which no L is actually required in the calculation, is the simplest method to be used with high accuracy. The accuracy of the 3-N solution is virtually independent of variations in L. For the other solutions with at least one pole equation applied, the accuracy depends upon the accuracy of L. Beam axes so calculated are generally accurate to 0.14° if L is obtained from K-pole separation, and to 0.7° if L is computed from K-pair separation.

APPENDIX D

CONVERSION OF U.S. CUSTOMARY UNITS TO SI UNITS

The International System of Units (designated SI) was adopted by the Eleventh General Conference on Weights and Measures in 1960. The units and conversion factors used in this report are taken from or based on NASA SP-7012, "The International System of Units, Physical Constants and Conversion Factors - Revised, 1969."

The following table expresses the definitions of miscellaneous units of measure as exact numerical multiples of coherent SI units, and provides multiplying factors for converting numbers and miscellaneous units to corresponding new numbers of SI units.

The first two digits of each numerical entry represent a power of 10. An asterisk follows each number that expresses an exact definition. For example, the entry "-02 2.54*" expresses the fact that 1 inch = 2.54×10^{-2} meter, exactly, by definition. Most of the definitions are extracted from National Bureau of Standards documents. Numbers not followed by an asterisk are only approximate representations of definitions, or are the results of physical measurements.

ALPHABETICAL LISTING

<u>To convert from</u>	<u>to</u>	<u>multiply by</u>
calorie (cal)	joule (J)	+00 4.184*
Celsius (C)	kelvin (K)	$t_K = t_C + 273.15$
Fahrenheit (F)	kelvin (K)	$t_K = (5/9)(t_F + 459.67)$
inch (in.)	meter (m)	-02 2.54*
kips f/in ² (ksi)	newton/meter ² (N/m ²)	+06 6.895
lbf/in ² (psi)	newton/meter ² (N/m ²)	+03 6.895
mil	meter (m)	-05 2.54*
torr	newton/meter ² (N/m ²)	+02 1.333

PREFIXES

The names of multiples and submultiples of SI units may be formed by application of the prefixes:

Multiple	Prefix
10 ⁻⁶	micro (μ)
10 ⁻³	milli (m)
10 ⁻²	centi (c)
10 ⁻¹	deci (d)
10 ³	kilo (k)
10 ⁶	mega (M)
10 ⁹	giga (G)